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JANUARY 1948

UNITED STATES
DEPARTMENT OF THE INTERIOR
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BUREAU OF MINES
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ANNUAL REPORT OF RESEARCH AND TECHNOLOGIC WORK ON COAL FISCAL YEAR 1947

BY

A. C. FIELDNER AND P. M. AMBROSE

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UNITED STATES DEPARTMENT OF THE INTERIOR - BUREAU OF MINES

ANNUAL REPORT OF RESEARCH AND TECHNOLOGIC WORK ON COAL

FISCAL YEAR 1947[1]

By A. C. Fieldner[2] and P. M. Ambrose[3]

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INTRODUCTION

This, the twelfth annual report of research and technologic work conducted by the Bureau of Mines on coal and coal products, summarizes the

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research and tests conducted from July 1, 1946, to July 1, 1947. The organic act of Congress establishing the Bureau of Mines authorized technologic investigations of all phases of mining, preparation, treatment, and utilization. These investigations[4] cover eight main fields of activity. The emphasis on individual projects varies from time to time. The United States possesses about one-half of the coal reserves of the world. These reserves are not always of the quality desired or in the place where they could best be used. It is necessary to continue research in order that increased output, greater recovery, and better quality can be maintained under safe mining conditions. Coal comprises 98 percent of the known fuel reserves of the United States and eventually must take over much of the requirements now supplied by petroleum and natural gas.[5]

The purpose of this report is to bring to the reader a resume of the work of the fiscal year. The material presented herein is based largely upon publications. In a few instances the results of research that would not otherwise be available in the immediate future are presented. For details concerning the research, it is suggested that the readers consult the publications given in the footnote references.

Collected references to all Bureau of Mines publications on coal are published in a series of bibliographies. Technical Paper 576 covers 1910 to 1935; Technical Paper 639 covers 1935 to 1940; and Technical Paper 698,[6] issued in 1947, covers 1940 to 1945.

SUMMARY

Following the unprecedented demand for coal during the war, the Bureau of Mines completed all war research on coal and directed its efforts toward improvements in mining, preparation, and utilization of coal for national security and peacetime economy. Special emphasis was given to conservation and safety research.

Service to Federal agencies was continued on fuel-utilization problems, including the sampling and analysis of coal, consulting on the purchase of fuel and fuel-burning equipment, tests of fuels and boiler-room equipment, instructions on the storage of coal, analyses of boiler water, recommendations as to boiler-water chemical treatment, and the supplying of boiler-water test kits. Such services, including those within the Bureau of Mines, required the analysis of 18,045 samples of coal, coke, char, tar, pitch, hydrogenated residues, heavy oils, pasting oils, organic compounds, distillation fractions, and coal-mine dusts. Government purchases and tipple

4/ Fieldner, Arno C., Coal-Research Activities of the Bureau of Mines: Bureau of Mines Inf. Circ. 7367, August 1946, 14 pp.

5/ Fieldner, Arno C., The National Fuel Reserves. Relation to the Future Supply of Liquid Fuels: Mech. Eng., March 1947, pp. 221-226 and 228.

6/ Fieldner, Arno C., Fischer, P. L., and Pollock, Marjorie B., Bibliography of Bureau of Mines Investigations of Coal and Its Products 1940 to 1945: Bureau of Mines Tech. Paper 698, 1947, 53 pp.

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inspections accounted for 3,666 samples; Bureau of Mines research on composition, preparation, combustion, carbonization, and liquefaction, including work for the Federal Geological Survey, accounted for another 7,713 samples. The coal-mine inspectors and engineers of the Health and Safety Branch submitted 6,623 samples of coal-mine dusts from the Nation's larger producing coal mines.

Increasing concern over depletion of high-grade coking-coal deposits led to the development of additional reserves of coal for making metallurgical coke. Two investigations to determine minable reserves of coking coals were completed, one in Maryland, where about 500,000,000 tons of coal that by suitable preparation methods could be used for making metallurgical coke were indicated, and one in Alabama, where a 14,000,000-ton reserve in thin beds was indicated. Another investigation was continued to determine the amount and coking quality of coal in Gunnison County, western Colorado, that could supply needed coking coal for the western steel industry. In Alaska, an investigation of bituminous-coal reserves was started in the Matanuska field.

The practicability of using scrapers and other light earth-moving equipment for strip-mining areas of limited extent and with moderate overburden was shown in a study completed this year. The efficacy of a scraper-shaker-loader designed by the Bureau was demonstrated, and minor improvements in design are being made before the machine is again placed in test operation. A special type of shearing machine was proved by underground tests to be particularly suited for cutting steeply pitching anthracite beds. Designs were completed and construction was started on a coal planer with vibrating blades to simultaneously shear off and load coal in longwall mining.

Factory tests and examination of tunnel air during construction of the Delaware aqueduct showed that the carbon dioxide, carbon monoxide, and oxides of nitrogen content of the air in Diesel exhausts did not increase to a prohibitive amount; also, the oxygen content of the air was not depleted to any significant extent.

Continuing its program to eliminate the hazards to life and property in the manufacture, storage, and use of explosives, the Bureau conducted over 2,300 tests on permissible explosives and blasting devices, new types of industrial explosives seeking to utilize surplus military stores, and hazardous chemical materials. Six new explosives were added to the permissible list, raising the total number approved to 186. To meet the requirements of increased mechanization in coal mines, a reevaluation of the Bureau's permissibility tests has been undertaken; and, after 650 large-scale tests in the Experimental mine, permanent approval was given for an increase in the maximum charge weight of permissible explosives from 1.5 to 3 pounds per shot.

Another approach to the problem of providing critically needed coking coal was followed in the Bureau's studies of upgrading high-sulfur or high-ash coals to make them suitable for metallurgical use. Such preparation studies

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were made in connection with the coal-investigation project in Maryland, where several hundred million tons of additional coking coal reserves are indicated, provided that the sulfur and ash can be reduced to a suitable figure. Limitations of present preparation methods to reduce sulfur content to meet metallurgical standards was shown in a study of coal from an area in western Pennsylvania estimated to contain half the recoverable coal in the Pittsburgh bed in this State. A systematic study of typical commercial preparation plants was initiated to appraise the factors that limit and influence the beneficiation of lower-grade coals, and field work at two plants has been completed. A method for recovering fine-size coking coal formerly wasted has been developed by the Bureau and is now in commercial use. Analysis of data obtained in a survey of coal-preparation practices in western Europe disclosed new ash-removal methods and technological developments that will be of value to the American coal industry. These included methods of preparing very low ash coals for the synthetic liquid-fuel industry and use of a cyclone coal washer for cleaning fine coal and clarifying washery water. Construction of the building to house the coal-preparation pilot plant at Bruceton, Pa., was completed, and equipment is being installed.

Research in subbituminous coal and lignite on a pilot scale indicated the efficacy of a steam-drying process that would expand the market area for these coals and permit their storage for long periods.

Types of fuel and fuel-burning equipment were determined for 38 new hospital projects of the Veterans Administration. At the request of the Navy, a program giving special instructions to Navy personnel on inspecting and sampling coal at Navy land stations was well-started, many stations being visited. Improvements were made in boiler-water conditioning for Federal boiler plants, and improved test procedures were devised for control of chemical treatment. Research was started on a new chemical for preventing corrosion in condensate return lines that gives promise of being much cheaper.

Research work was continued on corrosive external deposits on boiler equipment that cause excessive boiler outage and maintenance expense. One phase of this work has resulted to date in substantial improvement in the maintenance and operation of air preheaters throughout the United States.

Basic factors have been evaluated in the Bureau of Mines study of corrosion of boiler tubes. Field and laboratory studies of protective coatings to provide corrosion resistance are showing very promising results. Investigations of heat absorption and effect of coal-ash slag on operation of large central-station boiler furnaces was continued in laboratory and field studies. Eight tests of a gas producer designed to make rock wool and gas from Rhode Island anthracite have indicated the desirability of using preheated air for the blast. The volume of smoke produced by packaged fuels of various types was investigated in tests simulating domestic furnace conditions.

Aiding in the search for new coking coals to replace depleted reserves, the Bureau of Mines continued its investigation of the carbonizing properties

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of American coals, with tests on one coal each from Kentucky and Virginia, four from Pennsylvania, and five from Alabama. Similar tests were made on one coal from Venezuela and on three samples of asphaltic material from Argentina. Study of effect of coal size upon carbonizing properties indicated improvement of coking properties for the smaller sizes of some coals. Complete gasification of lignite was obtained by using an externally heated alloy retort for production of gas and char and fluidized-bed producer for gasification of the char.

The possibilities of efficiently using some of our coal resources without extensive underground work and of utilizing beds that cannot be mined economically has aroused Nation-wide interest in the underground gasification of coal. In January the Alabama Power Co. and the Bureau of Mines began an experiment with this method at Gorgas, Ala., where an isolated block of coal was ignited and gasified with various combinations of air, oxygen, and steam. The results of this exploratory test were favorable in that the coal was fully consumed in the gasified area and the roof rock expanded to occupy the space left as the coal was used up. However, the heating value of the gas was low. Further tests should be made.

Construction of the facilities required to carry on the Bureau's program of synthetic liquid-fuel research and demonstration has been hampered by wartime and postwar shortages of men and materials ever since the work was authorized in 1944. Nevertheless, three of the four major installations were completed or approaching completion at the end of the 1947 fiscal year.

Several new and improved methods that promise lower costs for gasoline and oil from coal are under development at Pittsburgh and at Bruceton, Pa., where new pilot plants are nearing completion.

A coal-hydrogenation demonstration plant to produce 200 barrels of gasoline a day is under construction at Louisiana, Mo. The site of this plant is the Missouri Ordnance Works, which was made available to the Bureau by the War Department.

Much information, unavailable during the war, on the mining, preparation, and utilization of coal in Germany and other European countries was widely disseminated to industry through distribution of published and microfilmed reports containing data gathered by Bureau of Mines personnel and other investigators while on foreign assignment.

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University of Alabama
University of North Dakota
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ORIGIN, COMPOSITION, AND PROPERTIES OF COALS

Inspection, Sampling, and Analysis

Coals, particularly those of high grade, continued to be scarce throughout the year owing, in part, to export demand and, in part, to domestic

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activity. As a result, Government agencies had difficulty in obtaining enough coal to meet requirements and in many cases had to either extend contracts made in previous years or accept such coals as were offered. The Office of Indian Affairs, whose requirements are almost entirely in the western fields, was able to purchase its coal supply on a guaranteed analysis basis. The Department of Justice purchased a part of its supply and the War Department purchased a limited part of its supply in the Sixth Army area on this basis. Other purchases were made at a fixed price not subject to adjustment for coal quality. Coal requirements of Federal agencies for the fiscal year were estimated at approximately 7,000,000 tons, the War Department being the largest purchaser and the Navy Department the second largest. Recommendations were made for awards of contracts for some agencies and furnished other agencies with analyses for use in evaluating coal bids and for use when substitute coals were offered.

Upon the request of the Navy Department, the Bureau instituted a program at Naval Stations for sampling coal and acting as technical advisor on its inspection and sampling. Two engineers, detailed for this work, made 150 visits to Naval Stations during which sampling procedures were set up and recommendations made for installing sampling equipment and for the storage and handling of coal.

For the use of various Government agencies in making coal purchases and for public information, tipple and face samples were collected at 160 mines in various States. Special samples for research on washability, carbonization, and gasification properties and miscellaneous tests were taken in 70 mines.

Analyses of Arizona, California, Idaho, Nevada, and Oregon Coals

The analyses of coals from five Western States - Arizona, California, Idaho, Nevada, and Oregon - have been published in a technical paper 7/ as another in the series describing coals of various States. Descriptions of the coal fields, including geology, occurrence, nature, and thickness of beds, were included and supplemented by maps showing location of beds in four of the States. Production, distribution, and use of coal in these States and the relationship of mine samples to commercial shipments were discussed. Descriptions of mine, tipple, and delivered samples included chemical analyses, calorific values, classification by rank, agglomerating indices, and fusing temperature of ash. The location and description of a number of operating mines were included, with data on nature of roof and floor and on thickness of coal and partings in the beds.

7/ Fieldner, A. C., Andrews, D. A., Hendricks, T. A., Huddle, J. W., Bell, C. H., Anderson, R. L., Snyder, N. H., Cooper, H. M., Abernethy, R. F., Tarpley, E. C., and Swingle, R. J., Analyses of Arizona, California, Idaho, Nevada, and Oregon Coals: Bureau of Mines Tech. Paper 696, 1947, 83 pp.

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Data on Coals in Districts 12, 14, and 16

A data book[8] giving information on rail shipping and truck mines having a daily capacity of 50 tons or more located in Coal-Producing Districts 12, 14, and 16, covering the bituminous-coal-producing areas in Iowa, Missouri, Kansas, Arkansas, Oklahoma, and Texas, was published. This book contained descriptive tables giving names of mines and producers, location, coal veins or seams worked, and originating railroads for those mines served by rail. Mines were indexed and their locations shown on maps. Tables of specimen analyses and of the usual range of analyses produced in the districts were given.

Laboratory Lists

A set of four lists[9] of commercial laboratories equipped to make various types of coal and coke analyses, determinations of free silica, and gas analyses was published. The Bureau of Mines does not make commercial analyses, and these lists of laboratories were compiled for the information and convenience of inquirers. They comprise partial lists of laboratories using methods recommended by the American Society of Testing Materials for analysis of coal and coke in the Eastern States (List 1), the Southern States (List 2), the Middle Western States (List 3), and the Western States (List 4).

Analyses were made of 3,666 coal samples from purchases of coal and tipple and breaker inspections taken in connection with Government fuel purchases. Of these, 71 percent were for the War and Navy Departments, and the remainder were for nine other departments and agencies. The Bureau's research and test work relating to the mining, preparation, and utilization of coal and coal products required analysis of 6,353 samples, and 360 samples of marine sediments from drill cores taken from the floor of the Gulf of Mexico were analyzed for the Geological Survey.

An increased number of coal-dust samples from a larger number of mines were required this year than in the previous year to assist the Federal coal-mine inspectors in determining whether safe conditions existed in mines and to assist in formulating recommendations in connection with mine inspections. In all, 6,623 samples of road, roof, rib, and gob dusts from 1,085 mines in 18 States were analyzed.

The analytical services given to aid the various research and service programs in the Bureau and to other Federal agencies required analysis of 18,045 samples and making 174,325 determinations.

8/ Fieldner, A. C., and Sweeny, H. P., Typical Analyses of Bituminous Coals Produced in Districts 12, 14, and 16: Bureau of Mines Data Book, vol. 7, 1946, 74 pp.

9/ Laboratory List 1, Eastern States, May 1946, 4 pp.; Laboratory List 2, Southern States, May 1946, 2 pp.; Laboratory List 3, Middle Western States, May 1946, 4 pp.; Laboratory List 4, Western States, May 1946, 2 pp.

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Constitution, Properties, and Analytical Methods

Extractable Waxes from Lignitic and Subbituminous Coals of the United States

The investigation to determine whether montan wax similar to that extracted commercially in Germany from certain brown coals can be obtained from low-rank coals of the United States was continued with extraction tests of lignitic and subbituminous coals from Arkansas, California; Colorado, Montana, North Dakota, Oregon, Texas, Washington, and Wyoming. Table 1 gives results of these tests, using benzene and a benzene-alcohol mixture, the solvents reported to have been used in Germany for extraction of montan wax from brown coal. The results show that the highest yields of extract were obtained from certain Arkansas and California lignites. The coals examined from the other sources do not yield enough extract to make them appear attractive as commercial sources of montan wax. Higher yields were obtained using the benzene-alcohol mixture than with benzene alone.

TABLE 1. - Yields of extract from air-dried coals, calculated to dry ash-free coal basis, percent

	Benzene	80 percent benzene, 20 percent ethyl alcohol
Lignitic coals		
Arkansas	8.8 - 10.8	13.6 - 16.6
California	8.2 - 9.9	15.1 - 15.3
Montana	-	2.6
North Dakota	1.5 - 2.2	2.9 - 4.5
Texas	2.1 - 2.3	5.6 - 5.7
Washington	2.7	4.4 - 5.7
Subbituminous coals		
Colorado	-	2.6
Montana	-	2.0
Oregon	-	3.0
Wyoming	-	2.9 - 5.4

Physical and chemical tests of the extracts from the Arkansas and California lignites examined showed that their properties were similar in many respects to crude commercial montan wax. The solvent used in the extraction had some effect on both yields and properties. The "resin" content of the extracts was in most cases somewhat higher than that reported in the literature for montan wax of commercial grade.

Analyses of Miscellaneous Materials

Chemical analyses were made of 123 samples of a wide variety of materials taken in connection with investigations concerned with the utilization of coal and health and safety in mines.

Twenty boiler-water compounds and 19 boiler scales or water-formed deposits were analyzed in connection with studies of boiler-water treatment

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in Government heating and power plants as part of the fuel-engineering service furnished by the Bureau of Mines to other Government agencies. Analyses were made of 10 samples of deposits from air heaters of boiler furnaces as part of a study of the properties of these corrosive deposits. Ten samples of external deposits from boiler tubes at several power stations and samples of iron pyrite and ferrous sulfide were analyzed in connection with an investigation of the mechanism of formation of corrosive deposits on boiler surfaces.

Analyses were made of 17 samples of slag and ash from tests of a slagging gas producer using Rhode Island anthracite as fuel for the direct production of mineral wool.

An analysis of ash of Takamatsu coal from Japan showed a composition somewhat similar to ash of some coals in the United States, but this ash contained a comparatively low Fe_2O_3 content of 4.1 percent.

Analysis of one soot remover showed it to be essentially common salt with a small quantity of magnesium sulfate and ultramarine blue coloring material, while another contained salt with some sodium chlorate and the same blue coloring material.

Eleven samples of rock-dusting materials were examined to determine their suitability for use in coal mines to prevent the propagation of coal-dust explosions. Limestone or dolomite dusts are preferred because they are generally low in silica and combustible matter, do not cake excessively when exposed to moisture, and are available in most coal fields of the United States. Gypsum dusts are seldom recommended, because they usually form a hard cake when wetted with water and dried, thus losing their effectiveness. Four of the samples examined were from Missouri and consisted of two limestones that would be suitable for rock dusting and two mixtures of limestone with sodium bicarbonate. The bicarbonate had been added on the theory that carbon dioxide would be liberated by heat in an explosion, creating an inert atmosphere that would add to the effectiveness of the rock dust. However, these mixtures when wetted with water and air-dried formed relatively hard cakes; therefore, they were not considered desirable for rock dusting. One sample of gypsum from Utah and three from Colorado showed high caking properties when wetted with water and were unsuitable for use in wet mines. One limestone from Colorado was of sufficient purity for rock dusting.

Five catalysts from coal-hydrogenation plants in Germany were analyzed for main constituents in connection with the Synthetic Liquid-Fuels Program. These analyses included determinations of silica, iron, aluminum, molybdenum, zinc, and chromium. Tin and total halogen were determined in three residues from coal-hydrogenation tests in which tin sulfide and iodoform had been used as catalysts. Tap water from the Synthetic Liquid-Fuel plant at Bruceton, Pa., was analyzed and found to be suitable for drinking, as it contained not more than 0.02 part per million of lead.

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Six samples of foundry coke from ovens in Alabama contained 0.022 to 0.041 percent phosphorus. An appreciable quantity of vanadium was found in a sample of coal from the Sillapata bed in Peru, South America. The ash of this coal contained 6.9 percent V_2O_5, which was equivalent to 0.95 percent in the coal, as received. Small quantities of vanadium also were detected in two other coals from Peru.

Analysis of a high-ash boghead-cannel coal from Quincy, Mo., proposed for use as a fertilizer material if high in potash, showed less than 1 percent potassium oxide. Three samples of experimental low-temperature coke made from coal to which sodium carbonate had been added to make activated coke were analyzed for carbon dioxide. Most of the carbonate remained unchanged during the coking period and accumulated largely in the finer sizes of coke. Fusain in a tipple sample of Hiawatha-bed coal from the King mine, Carbon County, Utah, was determined by the chemical oxidation method to be 1.7 percent on an as-received basis.

Other materials analyzed included a sample of roof shale from the underground gasification of coal project at Gorgas, Ala., a heat-retaining substance containing charcoal for covering ingots, a corrosion deposit from a Koehler lamp, copper sheet metal used in explosives research, sodium and gold chloride used as a standard in X-ray analysis, a liquid incendiary bomb that had been made from a light bulb, and furnace-black grit from a carbon-black plant in Texas.

Reflectance of Coal

A preliminary investigation was made of the method of quantitative petrographic analysis of coal proposed by Dr. C. A. Seyler in England and based on apparent discontinuous variations in reflecting power of the micro-constituents of coal. Seyler has set up a series containing nine steps of reflectance, ranging from 0.26 to 4.41 percent, and he states that the micro-constituents of coal, excluding spores and cuticles, have reflectance values varying discontinuously in the steps of this series, with the lowest values in vitrain and the highest in fusain. Approximately 200 reflectance determinations were made with a Berek microphotometer on each of two polished-coal blocks, one from a bright coal and one from a splint coal. Frequency graphs of the various observed reflectance values did not show maxima coinciding with Seyler's reflectance series. It was concluded that a more critical test of the Seyler method must await delivery of better equipment.

Electron Microscope Studies of Metal Fumes and Fischer-Tropsch Catalysts

The ability of the electron microscope to reveal characteristic crystalline shapes and sizes far beyond the power of the optical microscope was emphasized by study of several specimens. Figure 1 shows an electron micrograph of chromium fumes, examined along with other metal smokes in connection with health studies. There were extremely thin hexagonal crystals and very fine globular particles, as small as 20 angstroms (0.000002 millimeter) in diameter, very near the limit of resolution of the electron microscope.

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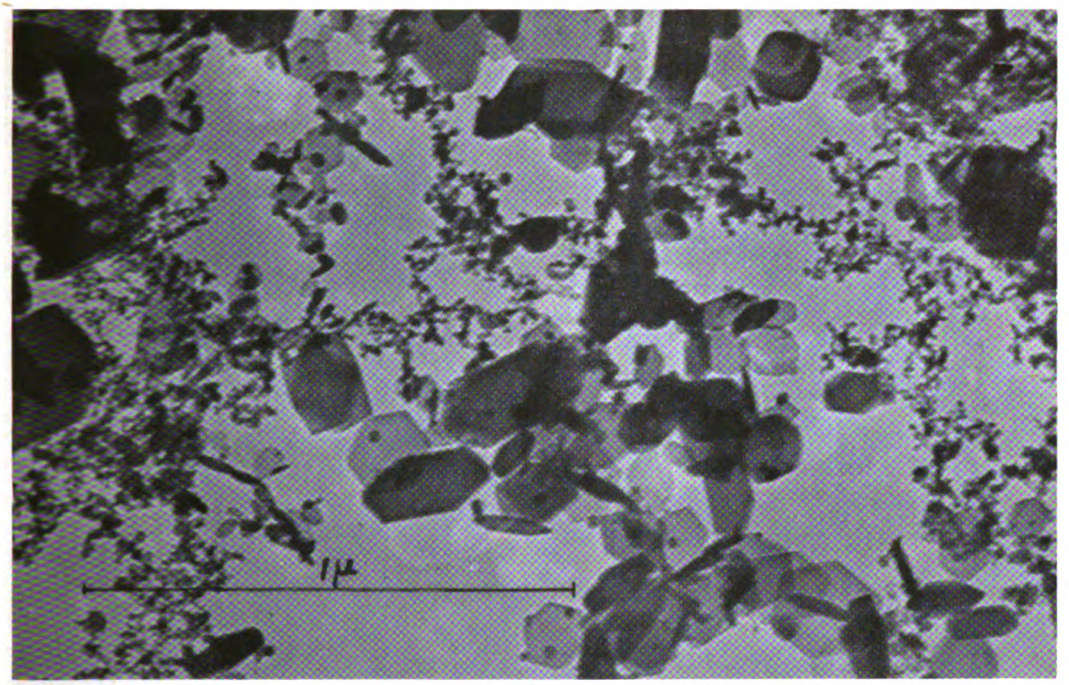

Figure 1. - Electron micrograph of chromium fumes.

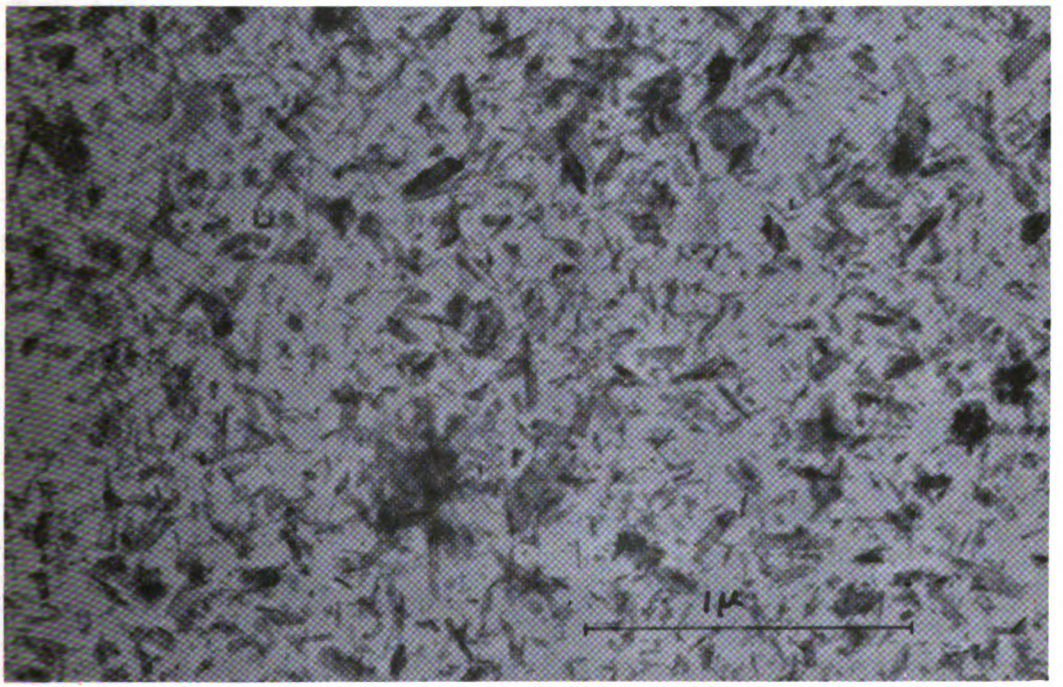

Figure 2. - Electron micrograph of copper oxide.

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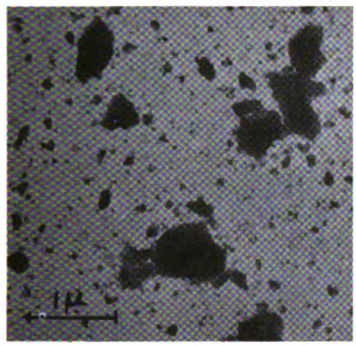

Figure 3.

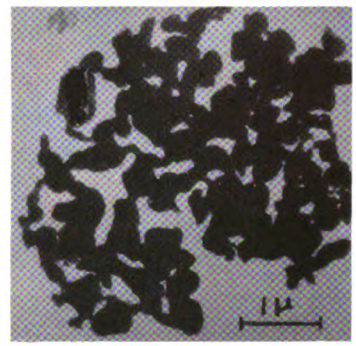

Figure 4.

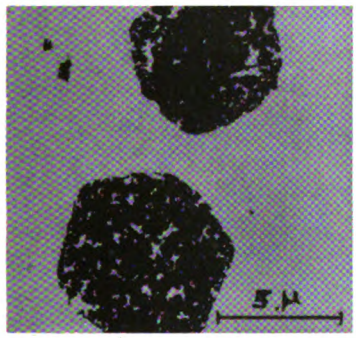

Figure 5.

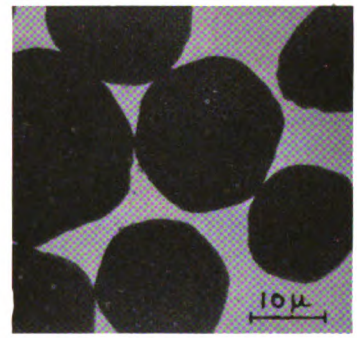

Figure 6.

Figure 3. - Electron micrographs of cobaltous oxide.

Figures 4-6. - Electron micrographs of cobalt metal reduced from cobaltous oxide, in various stages of dispersion.

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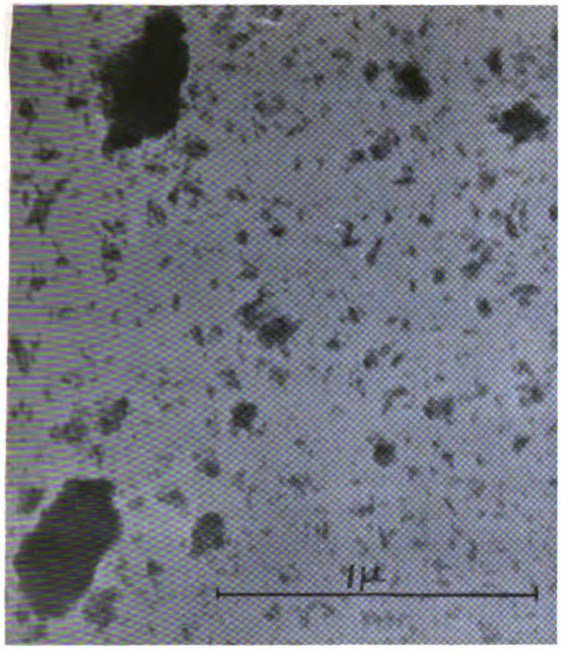

Figure 7. - Electron micrograph of cobalt basic carbonate.

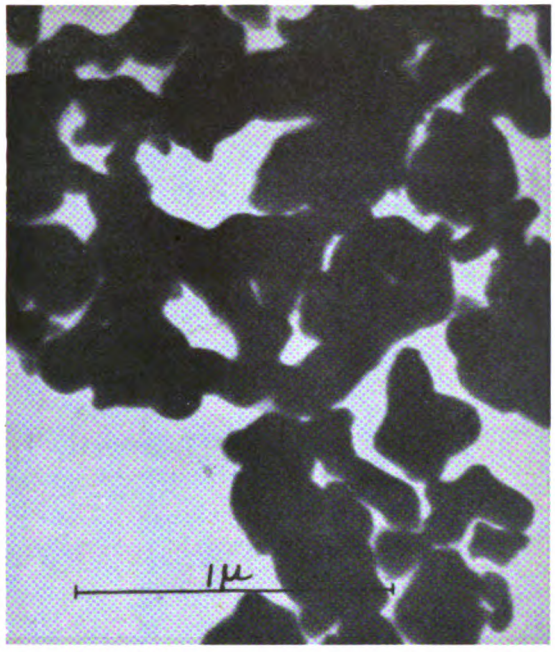

Figure 8. - Electron micrograph of cobalt reduced from cobalt basic carbonate.

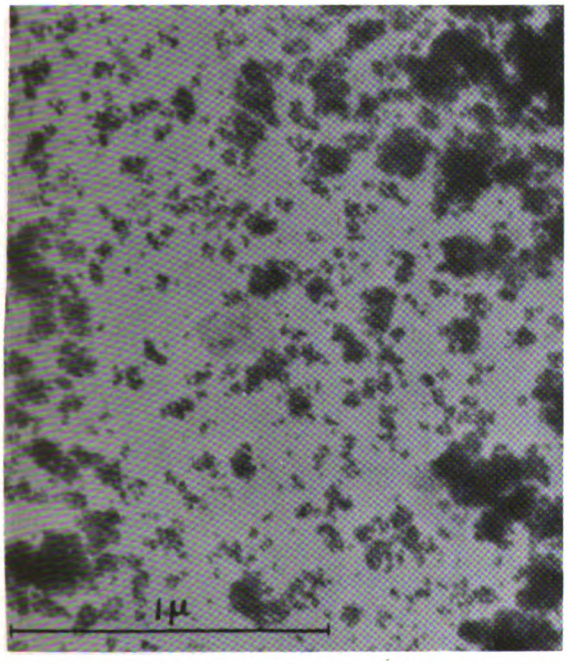

Figure 9. - Electron micrograph of cobalt reduced from cobalt basic carbonate in the presence of thoria and magnesia.

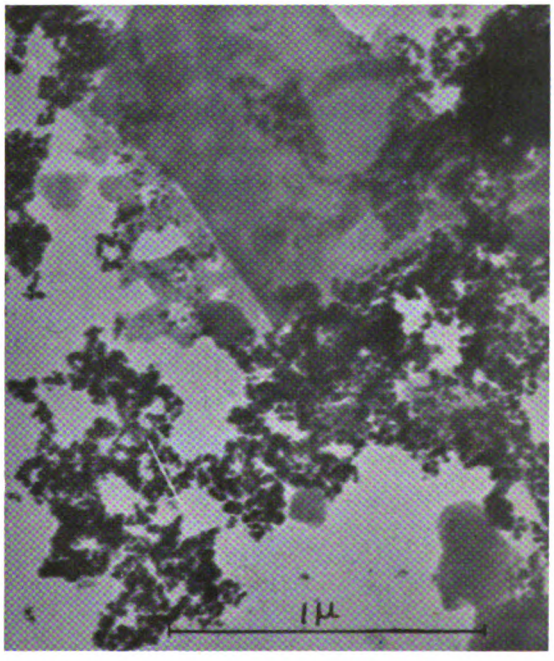

Figure 10. - Electron micrograph of cobalt reduced from cobalt basic carbonate in the presence of kieselguhr.

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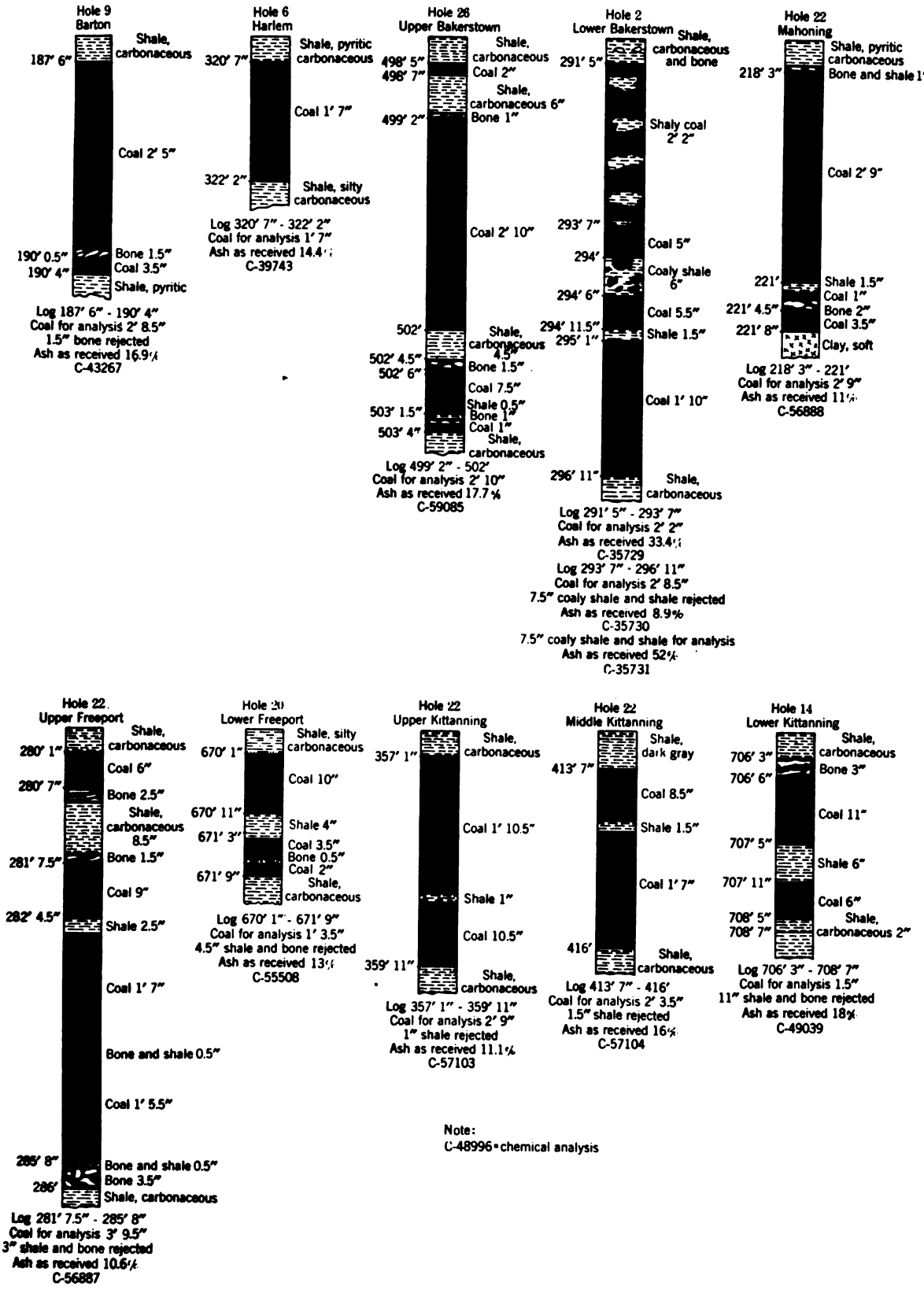

Figure 11. - Typical coal sections of drill cores from the Georges Creek Basin, Allegany and Garrett Counties, Md.

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Figure 2 shows a micrograph of copper oxide, used in catalysts for Fischer-Tropsch synthesis. The needle- or plate-like crystals are as small as 70 angstroms in length and 20 angstroms in width.

An interesting phenomenon was observed in cobalt metal powder reduced from cobaltous oxide used in Fischer-Tropsch catalysts. The oxide particles sintered into larger smooth droplets of cobalt that were aggregated into thin, hexagonal-shaped platelets. Figure 3 shows the oxide and figures 4, 5, and 6, various states of dispersion of the metal particles. The hexagons vary in diameter from 7 to 25 microns, and their thickness is apparently of the order of the diameter of the component particles. X-ray diffraction analysis also shows that the crystal phase present is hexagonal cobalt. These hexagonal aggregates may be formed by forces similar to those operating in normal crystal formation, but since the component particles do not lose their identity and the aggregates are easily fractured, they can hardly be classed as true crystals.

Electron micrographs have revealed the effects of the presence in catalysts of promoters, such as magnesia and thoria, and of carriers, like kieselguhr, in inhibiting the sintering of cobalt on reduction, thus preserving a large proportion of the surface area upon which the catalytic activity is so dependent. Figures 7 and 8 show the sintering effect when cobalt basic carbonate is reduced alone. This behavior is similar to that of the reduced cobaltous oxide except that hexagonal aggregation is not observed here. Figure 9 shows the much smaller particle size of the cobalt that results when the reduction is carried out in the presence of the promoters thoria and magnesia. Figure 10 shows that the addition of a kieselguhr carrier also decreases the size of the cobalt particles. Measurements on the micrographs give the following average particle sizes: Cobalt basic carbonate, 0.01 micron; reduced cobalt basic carbonate, 0.22 micron; reduced carbonate and promoters, 0.011 micron; reduced carbonate and kieselguhr, 0.028 micron. Surface area measurements by a nitrogen adsorption method confirm this beneficial behavior of promoters and kieselguhr.

Petrographic Studies of Coal

Petrographic analyses were made of selected portions from approximately 414 feet of cores from four test-drilling projects and the results used to correlate drill-log information and to selectively sample the coal for special chemical and physical tests. These cores were from the drilling projects at the Georges Creek field, Md.; Coal Creek area, Colo.; Coosa field, Ala.; and the Castleman basin, Md. Descriptive columnar sections which represent typical coal cores from the Georges Creek, Md., project are shown in figure 11.

Petrographic analyses were made of coal representing eight commercial coal beds from an 8-inch core recovered in test hole 5-33, Minnesota Creek area, near Paonia, Colo. Data showing depth, thickness, and petrographic composition of these coal beds are given in table 2.

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TABLE 2. - Petrographic analyses of eight coal beds in test hole 5-33, Minnesota Creek area, near Paonia, Colo.

Depth to coal bed		Thickness of coal		Percent			
Feet	Inches	Feet	Inches	Anthraxylon	Translucent attritus	Opaque attritus	Fusain
266	9	6	3	50.2	43.5	4.7	1.6
394	8	16	2	50.8	41.8	5.5	1.9
447	2	6	10	49.3	47.5	2.6	.6
458	6	4	11	54.2	43.2	2.1	.5
570	0	9	9	48.5	47.5	2.6	1.4
598	8	11	3	47.8	46.9	3.9	1.4
652	9	3	8	52.6	45.9	1.2	.3
670	3	3	1	59.3	38.6	1.6	.5
Total thickness of coal		61	11				

An assembled photograph of the split face of the core through the entire 16-feet 2-inch coal bed found at the depth of 394 feet 8 inches (see table 2) is shown in figures 12 and 13. The banded character of the coal can be readily seen by examining closely the photograph which shows a considerable surface of coal in clear detail.

Four column samples of bituminous coal from Illinois and Missouri and two samples of lignite were analyzed microscopically to determine the petrographic composition and evaluate these coals for use in hydrogenation tests. Significant data from the microscopic analysis are given in table 3. The relative amounts of anthraxylon plus translucent attritus and opaque matter plus fusain are important because it has been experimentally demonstrated that anthraxylon and translucent attritus give much higher yields in hydrogenation than the opaque matter and fusain in coal.

Petrographic analyses by means of particle identification and count under the low-power microscope were made of certain float-and-sink fractions prepared from Illinois No. 6 coal, Bankston Creek Collieries, Sahara No. 6 mine in connection with a coal-preparation study. The content of anthraxylon, bright attritus, dull attritus, fusain, and free mineral matter was determined in 16 float-and-sink fraction samples. The analyses showed a definite relationship between specific gravity of the float and the percentage of coal components present. Pertinent data from the analyses are presented in graphic form in figure 14.

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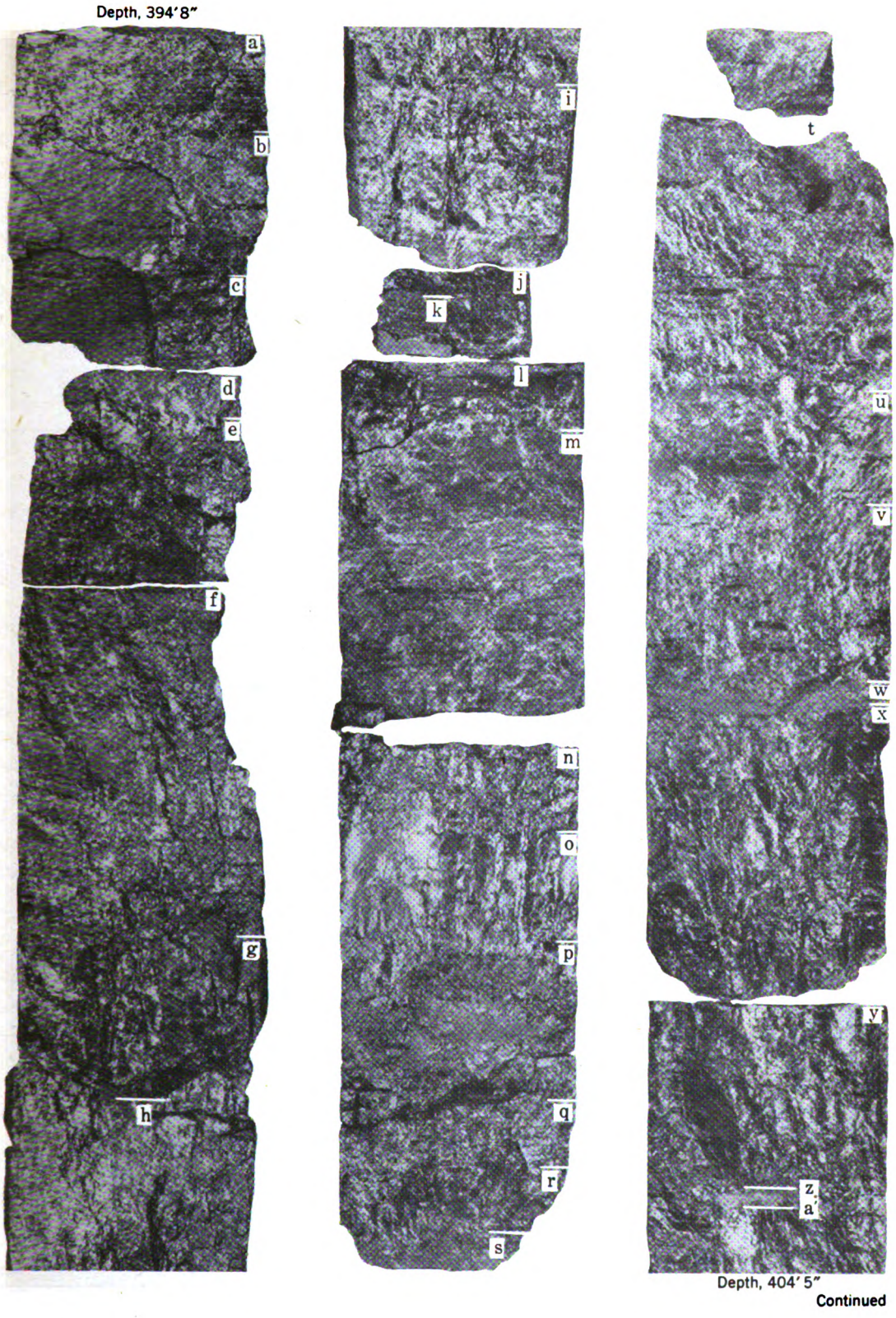

Figure 12. - 8-inch coal core split along natural fracture planes, 16 feet 2 inches thick, from drill hole 5-33, Paonia, Gunnison County, Colo. (See also figure 13.)

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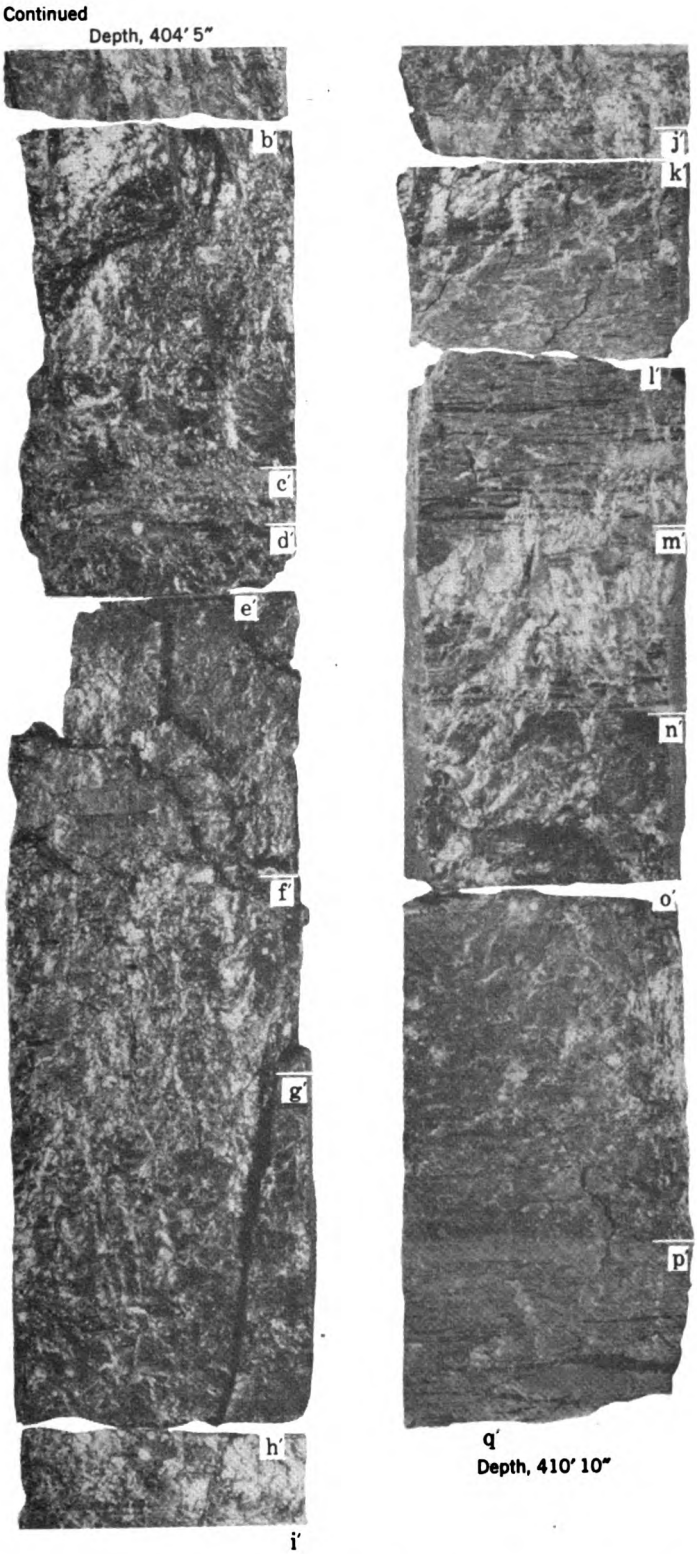

Figure 13. - 8-inch coal core split along natural fracture planes, 16 feet 2 inches thick, from drill hole 5-33, Paonia, Gunnison County, Colo. (Continuation of figure 12.)

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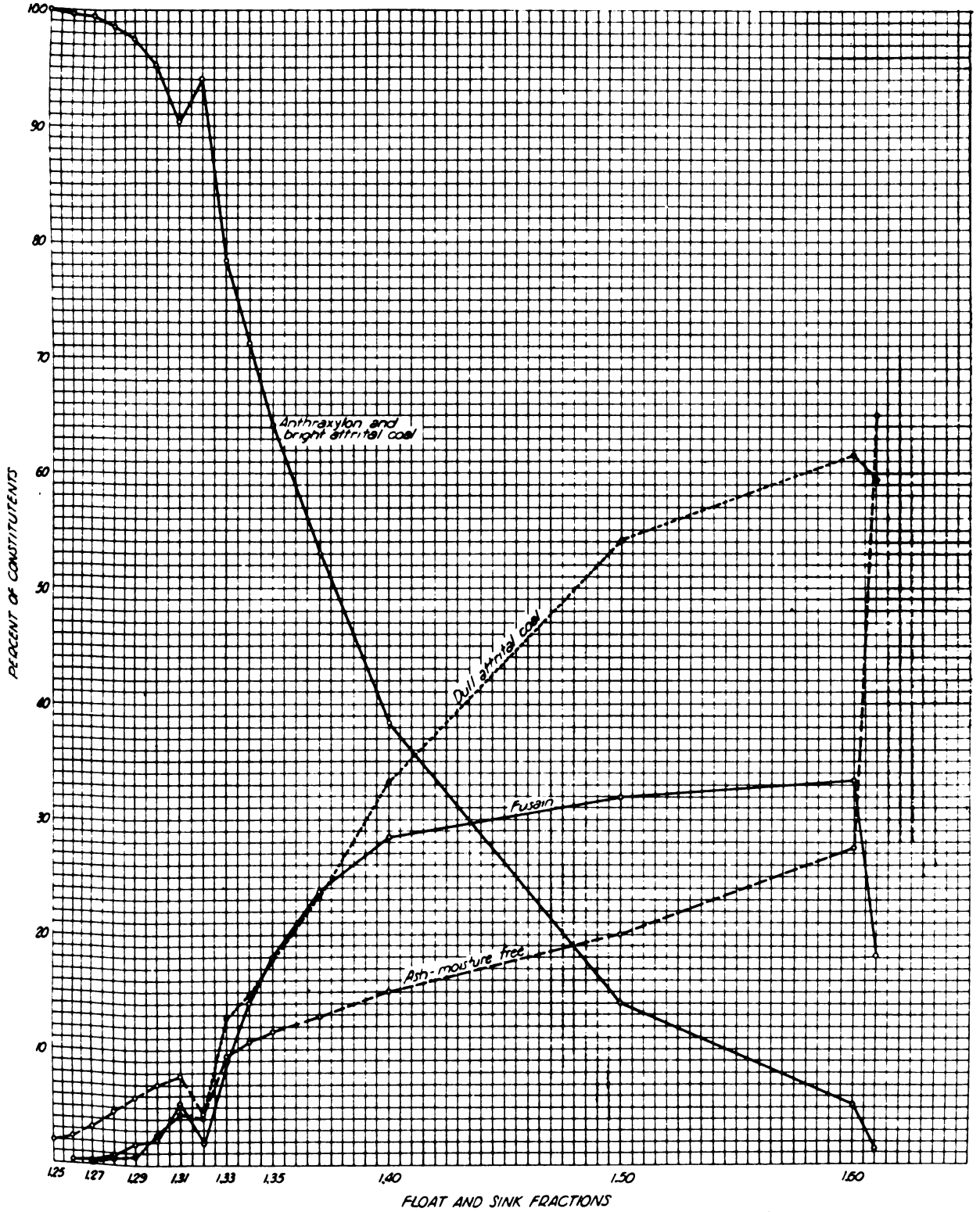

Figure 14. - Percentage of petrographic constituents of float-and-sink fractions, Sahara No. 6 mine, No. 6 bed, Illinois.

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TABLE 3. - Petrographic analyses of coal-column samples, percent

Petrographic components	No. 6 Coal Fulton Co., Illinois	No. 6 Coal Macoupin Co., Illinois	No. 5 Coal Sangamon Co., Illinois	Bevier Coal Macon Co., Missouri	Beulah Lignite Mercer Co., N. Dak.	Lehigh Lignite Stark Co., N. Dak.
Anthraxylon	77.2	74.7	72.2	60.5	53.4	59.6
Translucent attritus ..	17.2	15.7	20.5	30.6	25.1	36.0
Total anthraxylon plus translucent attritus	94.4	90.4	92.7	91.1	78.5	95.6
Opaque attritus	3.9	3.6	5.7	3.5	15.4	2.7
Fusain	1.7	6.0	1.6	5.4	6.1	1.7
Total opaque attritus plus fusain	5.6	9.6	7.3	8.9	21.5	4.4
Thickness of coal	4 ft. 2 in.	7 ft. 7 in.	5 ft. 2 in.	4 ft. 2 in.	10 ft. 9 in.	6 ft. 4 in.

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Glacial Peat Deposit near Pittsburgh

A peat deposit discovered near Bridgeville, Pa., a suburb of Pittsburgh, during strip mining of the Pittsburgh coal bed was examined.10/ This deposit of Pleistocene peat is unusual in its location, a considerable distance south of the area of glaciation, and it provides additional information regarding the geologic history of the region. Location of the deposit, the topography of the area, description of strata exposed in the stripping operation, and extent and depositional history of the peat were described. Fossil material identified includes mosses, ferns, coniferous seeds, insect remains, and bones of a fossil elephant, but a striking omission is the most common peat moss, Sphagnum.

Origin and Constitution of Coal

In response to many inquiries for summarized information on the origin and constitution of coal, the Bureau of Mines published as an information circular "What is Coal?".11/ This paper was originally delivered as a lecture by the late R. Thiessen before the Seventeenth Meeting of the Fuel Engineers of Appalachian Coals, Inc., at Cincinnati, Ohio, on January 11, 1937.

The botanical origin of the subbituminous coal of the Coos Bay field in southwestern Oregon was discussed in a paper presented before the Paleobotanical Section of the American Botanical Society at Boston, December 26, 1946, and later published in the American Journal of Botany.12/

The unusual uniformly bright luster of the Coos Bay coal results from the large amount of finely divided woody tissue accompanying the coal. Other features of the coal revealed by microscopic study were abundance of plant microfossils, particularly fungi of the sclerotia type, and the virtual absence of fusain.

Study of Materials Relating to Acid Formation in Coal Mines

In connection with studies relating to acid formation in coal mines, four concretions, supposedly of the sulfur-ball type, were obtained. The minerals in these concretions were determined by petrographic and X-ray diffraction methods to provide a better basis for understanding their origin. The following minerals were identified: Analcite, brammallite, calcite,

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10/ Schopf, James M., and Cross, Aureal T., A Glacial Peat Deposit Near Pittsburgh: Am. Jour. Science, vol. 245, No. 7, July 1947, pp. 421-433.

11/ Thiessen, Reinhardt, What is Coal? Bureau of Mines Inf. Circ. 7397, June 1947, 53 pp.

12/ Schopf, James M., Botanical Aspects of Coal Petrology; Coal from the Coos Bay field in Southwestern Oregon: Am. Jour. Botany, vol. 34, No. 6, June 1947, pp. 335-345.

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dolomite, kaolinite, melanterite, and pyrite.[13] A discussion on the occurrence and formation of analcite is included. The presence of analcite indicates that waters penetrating the joint cracks of the juvenile coal bed were alkaline in their reaction.

COAL MINING

Experimental Mine and Dust Explosions

Demonstrations

Educational demonstrations of the hazards of mine explosions and of means for their prevention were continued during the year for the benefit of workers and officials of the coal-mining industry, and for mining students. Two large public demonstrations, attended by 1,500 people, and many smaller ones were given at the Experimental coal mine. The second large demonstration, which was held a few weeks after the Centralia coal-mine explosion, aroused Nation-wide interest. A new feature that was demonstrated was the violent character of the explosion of a natural gas-air mixture in a mine entry with the mixture containing only 90 cubic feet of natural gas.

In connection with two large safety meets held by mining groups in Kentucky, a member of the staff gave several coal dust explosion demonstrations and exhibited the inhibiting effect of rock dust.

A color motion picture of one of the large Bruceton demonstrations was shown at the annual exhibit of the American Mining Congress in Cleveland, Ohio. Motion pictures of the demonstrations were also shown in newsreels throughout the country.

Technical Assistance and Services to Others

Continued assistance was given in the preparation of safety codes on dusting explosion prevention and on explosion pressure release by committees of the National Fire Protection Association, and in the preparation of safety digests by the National Safety Council. Technical consulting advice was also rendered on dust-explosion prevention and other miscellaneous safety problems to the War Department, the Department of Agriculture, the National Advisory Committee for Aeronautics, State labor departments, insurance associations, and numerous manufacturers.

Coal was mined from the Experimental mine and prepared for studies on hydrogenation, carbonization, and combustion by the Coal and Synthetic Liquid Fuels Divisions. The mine was made available to the Safety Division for investigating underground communication systems and for studies of trip lights on mine cars. During the year about 100 technical men, including foreign guests from many countries, visited the laboratories and the mine to become acquainted with the research facilities and test procedures.

13/ Foster, Wilder D., and Feicht, Florence L., Mineralogy of Concretions from Pittsburgh Coal Seam, with Special Reference to Analcite: Am. Mineral., vol. 31, Nos. 7 and 8, July-August 1946, pp. 357-364, incl.

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Coal Investigations

Coal Creek, Gunnison County, Colo.

Additional sources of coking coal west of the Continental Divide are needed for western steel plants; and as the analyses of coal obtained in diamond-drill cores in a hole drilled in Gunnison County, Colo., at the confluence of Anthracite Creek and the North Fork of Gunnison River indicated that these coals had coking qualities, a reconnaissance was made of the area adjacent to Anthracite, Muddy, Snowshoe, and Coal Creeks. This area is being investigated by diamond drilling.

The interest shown by coal-mine operators in the West in additional reserves of coking coal has made it advisable to publish the preliminary results of the investigation.14/

The area investigated comprises secs. 4, 7, 8, 9, 10, 11, 15, 16, 21, 22, 23, and 28, T. 13 S., R. 89 W., Gunnison County, Colo. The coal and mining rights in most of the land in the area are owned by the Government. The center of the area is approximately 6 miles east of the end of the Denver & Rio Grande Western standard-gage line to Somerset and the Oliver mine.

The topography of the area is mountainous and is traversed by several streams, which unite to form the North Fork of the Gunnison River. Altitudes range from 6,200 feet in the valleys to 8,000 feet on the plateaus. Marcelina and West Beckwith Mountains, east of the area, rise to altitudes over 12,000 feet.

The coal beds occur in the Mesaverde formation of upper Cretaceous age. The coal-bearing members comprise sandstone, shale, and coal beds. The coals, which are bituminous in rank, occur in the strata above the Rollins sandstone. The prevailing dip is approximately 5 percent or 3° northeast.

To date, 18 diamond-drill holes to obtain minimum 2-1/8-inch cores have been drilled to determine the thickness, physical characteristics, and extent of the coal beds and the character of the overlying and underlying strata. One hole to recover 8-inch cores of the coal beds was drilled to obtain enough coal for carbonization tests. The locations of the holes are shown in figure 15, and a condensed log of hole 8-9 follows:

14/ Toenges, Albert L., Turnbull, Louis A., Davis, J. D., and Reynolds, D. A., Investigation of Coal Deposits in the Coal Creek District, Gunnison County, Colo. - Progress Report 1: Bureau of Mines Rept. of Investigations 4104, 1947, 20 pp.

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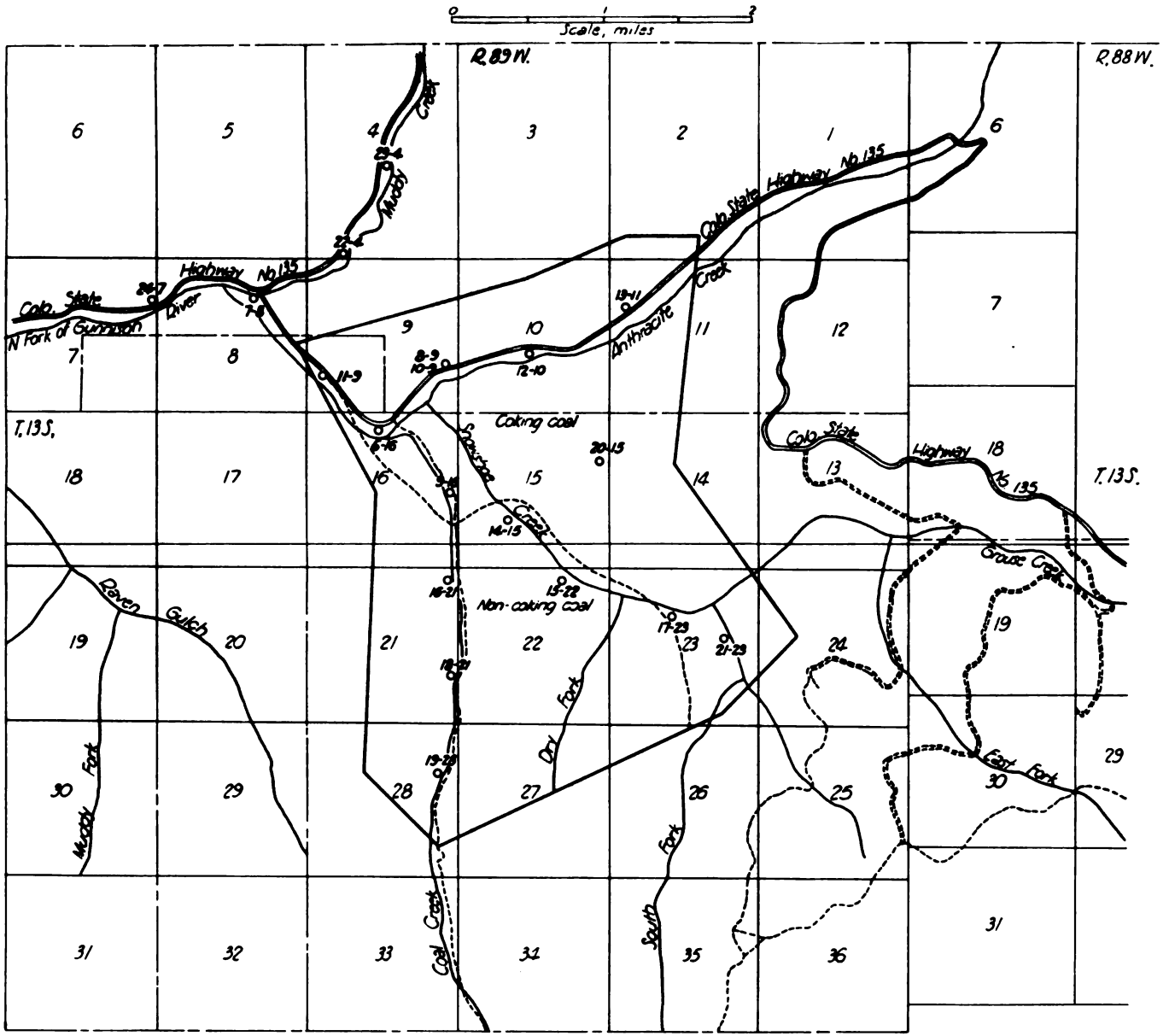

Figure 15. - Diamond drilling in Coal Creek area, Gunnison County, Colo.

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Hole 8-9

Location: 1,500 feet N. and 600 feet W. of SE. corner, sec. 9, T. 13 S., R. 89 W.
Surface elevation: 6,447 feet.

Depth From		Depth To			Thickness		
Feet	Inches	Feet	Inches	Material	Feet	Inches	Remarks
0	0	908	0	Shale, sandstone, and thin coal beds.	908	0	
908	0	929	3	Medium sandstone containing carbonaceous material.	21	3	
929	3	933	9	COAL.	4	6	
933	9	934	8-1/2	Carbonaceous shale.		11-1/2	
934	8-1/2	934	9-1/2	Bony coal.		1	
934	9-1/2	942	6	Carbonaceous, sandy shale.	7	8-1/2	
942	6	943	0	Bone.		6	
943	0	950	0	COAL.	7	0	
950	0	952	7-1/4	Mottled, gray, silty shale and carbonaceous shale.	2	7-1/4	
952	7-1/4	953	8-3/4	Carbonaceous shale.	1	1-1/2	
953	8-3/4	954	2-1/4	COAL.		5-1/2	
954	2-1/4	955	11-1/4	Carbonaceous shale.	1	9	
955	11-1/4	962	6	COAL.	6	6-3/4	
962	6	963	2	Sandy bone.		8	Top of Rollins sandstone 963 feet.
963	2	972	9	Medium to coarse sandstone with plant casts and scattered streaks of carbonaceous material.	9	7	
972	9	1003	0	Medium sandstone with abundant halymenites.	30	3	

The chemical analyses of the coal beds penetrated in hole 8-9 are given in table 4.

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TABLE 4. - Analyses of coal cores, Coal Creek, Gunnison County, Colo.

Lab. No.	Drill hole	Conditions[1]	Proximate, percent			Ultimate, percent						Calorific value, B.t.u.	Ash-fusion temperatures, °F.[2]	Agglutinating index[3]
			Moisture	Volatile matter	Fixed carbon	Ash	Sulfur	Hydrogen	Carbon	Nitrogen	Oxygen			
C-50797	8-9	1	2.7	40.0	53.8	3.5	.8	5.8	78.0	1.7	10.2	14,030	2,180 IDT	5.2
929' 3"-933' 9"		2	-	41.1	55.3	3.6	.8	5.6	80.1	1.8	8.1	14,420	2,420 ST	
		3	-	42.6	57.4	-	.9	5.8	83.1	1.8	8.4	14,960	2,620 FT	
C-50757	8-9	1	2.3	39.7	52.0	6.0	.5	5.6	76.4	1.7	9.8	13,690	2,210 IDT	5.8
943' 0"-950' 0"		2	-	40.7	53.2	6.1	.5	5.5	78.2	1.7	8.0	14,020	2,310 ST	
		3	-	43.3	56.7	-	.6	5.8	83.3	1.9	8.4	14,930	2,600 FT	
C-50758	8-9	1	2.3	38.3	51.4	8.0	.6	5.5	74.6	1.6	9.7	13,350	2,800 IDT	5.2
955' 11-1/4 - 962' 6"		2	-	39.2	52.7	8.1	.6	5.4	76.4	1.7	7.8	13,670	2,850 ST	
		3	-	42.7	57.3	-	.7	5.8	83.1	1.8	8.6	14,880	2,910+ FT	

1/ 1. Sample as-received; 2. Dried at 105° C.; 3. Moisture- and ash-free.
2/ IDT - Initial deformation temperature; ST - Softening temperature; FT - Fluid temperature.
3/ Ratio silicon carbide:coal, 15:1, crushing strength in kilograms.

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The results obtained in diamond drilling show that the minable coal beds exist near the base of the coal-bearing formations immediately above the Rollins sandstone. Three coal beds were found to be reasonably persistent at this horizon. The Upper bed, in secs. 9, 10, and 11 and the north half of secs. 15 and 16, ranges from 3 feet 9 inches to 5 feet 3 inches in thickness. In other parts of the area, this bed apparently is not minable. The Middle bed is fairly persistent at minable thickness throughout the area. It ranges in thickness from 4 feet to 10 feet 3 inches, except in hole 9-16, where it is only 1 foot 10 inches thick. However, drilling indicated that this bed is part of the Lower bed in some places, as shown in the logs of holes 14-15, 15-22, 17-23, and 21-23. The Lower bed is the most persistent in thickness and extent throughout the area. The thickness of this bed ranges from 6 feet 4 inches to 14 feet 10 inches, except in holes 16-21, 18-21, and 19-28, where the bed is very thin. These three holes are on Coal Creek, at the southern end of the area drilled, and it is evident that the Lower bed is not present at minable thickness in this particular area.

The additional work to be done in the Coal Creek district will furnish more data regarding the thickness and continuity of these three beds.

Preliminary estimates of measured reserves of coal in the area tested by drilling to date, are as follows:

	Tons
Coking coal	65,000,000
Noncoking coal	35,000,000
	100,000,000

The recovery in mining should range from 70 to 80 percent of the total reserves.

The first hole drilled at this project (6-16) encountered a small amount of oil and a strong flow of gas at a depth of 400 feet, about 250 feet above the upper minable coal bed. Samples of the oil were analyzed at the Bureau of Mines Petroleum and Oil-Shale Experiment Station, Laramie, Wyo. The results of those analyses show the oil to be a low-gravity, naphthenic oil, which contains no gasoline or kerosine and has no lubricating qualities. Evidently, the oil has little commercial value except as cracking stock in a refinery.

Gas was found in most of the holes, and the quantities and pressures were especially large in hole 19-28 and other holes on Coal Creek. The gas apparently is present in several hundred feet of strata above the coal beds, as well as at the horizon of the coals, and must be considered in planning mining operations.

A strong flow of water also was found in hole 19-28 at about the horizon of the coal beds. No doubt, the intensity of this flow was increased by the pressure of the gas in the hole. Water was found in other holes, but not in amounts that would affect mining operations adversely.

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The cores from hole 10-9 were large enough to be carbonized by the Bureau of Mines-American Gas Association (BM-AGA) method, using 13-inch-diameter cylindrical retorts of about 85 pounds capacity. Table 5 gives the yields of carbonization products at 900° C., and table 6 gives the physical properties of the cokes. Corresponding results for Sunnyside coal, Sunnyside mine, Carbon County, Utah, are included in both tables for comparison.

The drilled samples yielded more coke, gas, and tar and less liquor than the Sunnyside coal, although the differences were small. The Lower bed (286) gave the highest yield of coke (65.2 percent); the Middle bed (285) yielded the most gas (18.1 percent); and the Upper bed (284) gave the highest yield of tar (7.5 percent). Differences in the yields of liquor, ammonium sulfate, and light oil probably are not significant.

The quality of the cokes obtained from the drilled samples may be judged by comparing their strength-test indexes with those of the coke obtained from Sunnyside coal under identical test conditions. The coking properties of Sunnyside coal are well-known, and the sample used in this study was representative of the bed. These data indicate that the Upper and Lower beds coke more strongly than the Sunnyside bed. The Middle bed yielded weaker coke than the Upper and Lower beds; it was slightly inferior to Sunnyside coke. Technologists of the Columbia Steel Co. judge their coke largely by the proportion that remains on the 1/2-inch screen in the tumbler test, which, in their opinion, should be 80 percent or more. By that standard the cokes from the Upper and Lower beds would qualify as metallurgical grade.

The chemical composition of the cokes from all three beds was satisfactory. The cokes from the Upper, Middle, and Lower beds contained 6.3, 8.3, and 10.9 percent ash and 0.9, 0.5, and 0.4 percent sulfur, respectively.

Additional diamond drilling will be done and carbonization tests made on coal in another part of the area. Final estimates of reserves and conclusions as to the coking quality of the coals cannot be made until the investigation is completed.

Coking-Coal Deposits on Lookout Mountain, DeKalb and Cherokee Counties, Ala.

An area comprising about 140 square miles on southern Lookout Mountain in DeKalb and Cherokee Counties, Ala., was investigated for coking coal.15/ Six coal beds 18 inches or more in thickness are represented in this area, but this project was confined principally to the third bed, correlated as the Upper Cliff No. 1 seam, and indicated by preliminary investigation as being a low-volatile, high-grade coking coal. In all, 95 holes were drilled. The very friable nature of the coal made it difficult to recover complete cores; but the coal was found to have good coking qualities, making it suitable for blending with high-volatile coking coal for the production of metallurgical coke.

15/ Coulter, Don M., Coking-Coal Deposits on Lookout Mountain, DeKalb and Cherokee Counties, Ala.: Bureau of Mines Rept. of Investigations 4030, 1947, 89 pp.

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TABLE 5. - Yields of carbonization products, as-carbonized basis

Coal no.	Description	Yields, percent by weight of coal							Yields per ton of coal			
		Coke	Gas	Tar	Light oil	Free ammonia	Liquor	Total	Gas, cubic feet	Tar, gallons	Light oil, gallons in gas	$(NH_4)_2SO_4$, pounds
284	Upper core	64.2	17.4	7.5	0.85	0.124	7.7	97.8	10,800	15.7	2.35	21.7
285	Middle core	64.5	18.1	6.7	.95	.128	7.0	97.4	11,100	13.8	2.62	20.4
286	Lower core	65.2	17.3	7.2	.87	.120	7.3	98.0	10,500	15.0	2.43	20.9
276	Sunnyside	64.1	17.0	6.4	.93	.101	8.6	97.1	10,550	13.2	2.54	16.4

TABLE 6. - Physical properties of coke, determined by Columbia Steel Co. methods

Coal no.	Description	Shatter test, cumulative percent, upon -				Tumbler test, cumulative percent, upon -			
		1-1/2-inch screen	1-inch screen	3/4-inch screen	1/2-inch screen	1-1/2-inch screen	1-inch screen	3/4-inch screen	1/2-inch screen
284	Upper core	51	81	88	94	22	62	71	80
285	Middle core	28	70	80	91	19	53	64	76
286	Lower core	37	80	89	96	25	65	76	84
276	Sunnyside	43	75	84	92	20	55	65	76

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Georges Creek Basin, Md.

The annual production of coal from mines in the Georges Creek field in Allegany and Garrett Counties, Md., has declined from 2,250,000 before 1931 to about 1,800,000 tons in the past few years. The greater part of this production was from mines working in the upper coal beds /Pittsburgh (Big Vein) and Sewickley (Tyson)/, which are approaching depletion after many years of mining. The annual production of the field is certain to decline further with an accompanying loss of employment for miners, unless production from the lower, thinner beds can be increased.

The coals of the lower beds are low-volatile coking coals, some of which are suitable for blending in the manufacture of metallurgical coke. Another important use for this semismokeless coal is for domestic and industrial use in the nearby Washington, Baltimore, and Pittsburgh areas. Little was known of the continuity and thickness of these beds, and the purpose of this investigation was to determine the minable reserves, thickness, physical characteristics, and chemical properties of these coals with the objective of obtaining necessary data for the development of mines in these lower beds. The field investigation has been completed, and data are being assembled and will be published.

Coosa Coal Field, Alabama

Coal mining in the Coosa Coal field, Alabama, has been conducted intermittently for many years. This field, which is east of the Warrior and Cahaba coal fields, is traversed by the main line of the Seaboard Airline Railway and adjacent to the proposed Coosa River development.

Reserves of coking coal in the Warrior field have been seriously depleted, and coals from the Coosa field might prove to be an additional source of metallurgical coke for southern steel plants. Information regarding the continuity and character of the coal beds in the Coosa field was too meager to encourage the development of modern mechanized mines, and this investigation was undertaken to determine the thickness and extent of the coal beds, physical conditions in and surrounding the beds that would influence mining, and the chemical and washability characteristics of the coals.

A reconnaissance of the area was made in the summer of 1945 by Bureau of Mines engineers and geologists of the Federal Geological Survey. Following this reconnaissance, the investigation of the field was undertaken. Operating and abandoned mines in the field, where accessible, were examined, and all maps and other available information regarding abandoned mines were secured. Sections of the Hammond, Brewer, Coal City, and Fairview beds were measured in abandoned and operating mines and in test pits, which were excavated at the outcrop of the Fairview bed. Face samples for petrographic study and chemical analysis were secured at mines in the Hammond, Brewer, Coal City, and Fairview beds. A 1-1/2-ton sample was obtained from the abandoned Soot Creek mine (Fairview bed) for washability tests at the Southern Experiment Station, Bureau of Mines, Tuscaloosa, Ala. The thickness of the

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beds and the physical conditions in and surrounding these beds were determined from cores of 24 diamond-drill holes. Diamond-drill cores of the coal beds also were used for petrographic examination and chemical analyses.

The field investigation was completed; and the results of the work, including estimated reserves, petrography, and chemical properties of the coals and the washability characteristics of coal from the Fairview bed, will be published.

Castleman Coal Basin, Md.

A source of low-volatile, semismokeless coal, especially desirable for use as smokeless fuel in cities and for blending with high-volatile coal for making metallurgical coke, is indicated in the Castleman Basin of Maryland. Because of the nearness of this field to users of low- to medium-volatile coal, an investigation of the field has been undertaken to determine minable reserves of coal in the area. Development of modern, mechanized mines cannot be undertaken until the reserves are established.

Deep River, N. C.

Investigation of the Deep River coal field was begun in June 1947 and is in progress. Additional diamond drilling is being done to determine the minable reserves of coal in this field. Preliminary tests of the coal indicate that it has coking properties.

This coal field is situated in a center of population which now is supplied principally with fuel from the distant West Virginia and Virginia coal fields. The cost of this coal is necessarily high. Should the investigation prove minable reserves sufficient for the development of a modern mine, fuel costs in this area should be reduced materially.

The area being investigated in the Deep River field comprises approximately 1,250 acres.

Alaska

An area of bituminous coal in Alaska, east of Eska Creek adjacent to the abandoned Eska mine had been studied by geologists of the Federal Geological Survey. This study indicated that physical conditions were favorable for the development of a modern mechanized mine should minable reserves of coal be proved by diamond drilling. This area is accessible to the surface plant and washery of the Eska mine, and these facilities could be used if the investigation by diamond drilling proved a reserve of minable coal. Diamond drilling in this area was in progress at the end of the fiscal year. The reported life of the Evan Jones mine, which is the only operating bituminous mine in Alaska, is 3 years, and should the investigations in the Eska Creek area develop a mine site, an operation here could supply bituminous coal until the minable reserves in the Wishbone Hill area can be established, and the possibility of developing a large modern mine in this area determined.

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Mining Methods and Practices

Strip Mining of Bituminous Coal

The urgent need for coal to supply industry, which expanded rapidly during the war, accelerated the use of scrapers and light earth-moving equipment in bituminous-coal strip mining. The coal industry experienced a severe shortage in manpower and difficulty in obtaining equipment for new underground mines and replacements for worn-out machinery in operating mines. Strip mining requires fewer man-hours per ton of production than underground mining in similar beds, and the time for developing a strip mine is shorter than that required for developing an underground mine of the same capacity. In Maryland, Pennsylvania, and West Virginia, the production of strip-mined coal increased between 1941 and 1945. In Maryland the increase was from 0.7 percent to 11.6 percent of the total bituminous-coal production in the State. The increase in Pennsylvania was from 6.5 percent to 20.6 percent and in West Virginia from 0.8 percent to 9.3 percent.

In recent years there has been rapid progress in the technique of strip-coal mining. Introduction of the use of scrapers and other light earth-moving equipment in strip mining of relatively small areas of coal in mountainous terrain is one of the recent advancements in the practice of coal strip mining and is responsible for the recovery of many small areas of coal that could not be mined economically in any other way. Coal beds underlying many of these areas cannot be mined by underground methods because of the shallow overburden and the difficulty of supporting the weak strata overlying the coal. Reserves of coal in place in these areas usually are not sufficient to justify the large initial cost of large shovels or draglines.

The operations at one mine in Maryland, one mine in Pennsylvania, and four mines in West Virginia using tractor-scrapers and other light earth-moving equipment were studied.16/

The areas at most of the mines were explored, by the owners, by diamond drilling, trenches dug by bulldozers, or hand-dug test pits before stripping was started. This exploration determined the location of the outcrop and the thickness and character of the overburden and also located the limit of weathered coal. The overburden moved at the mines studied ranges from a few feet to a maximum of 60 feet of soil, clay, shale, and limestone.

Tractor-scrapers ordinarily are used to move the overburden in outcrop and contour stripping in areas where the topography is rolling or hilly and favorable for the disposal of overburden in spoil piles entirely removed from the strip mine. The portion of the overburden moved by these units ranges from 12 to 50 feet of soil, clay, and shale. At two mines, small stripping shovels also were used in conjunction with tractor-scrapers.

16/ Turnbull, Louis A., Shields, Joseph J., Dowd, James J., Fish, Edward L., and Toenges, Albert L., Use of Scrapers and Other Light Earth-Moving Equipment in Bituminous-Coal Strip Mining: Bureau of Mines Rept. of Investigations 4033, 1947, 37 pp.

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Figure 16. - Loading scraper, tractor pulling, bulldozer pushing, West Virginia.

Figure 17. - Tractor-scraper digging shale. Tractor in background moving into position to push unit while loading, West Virginia.

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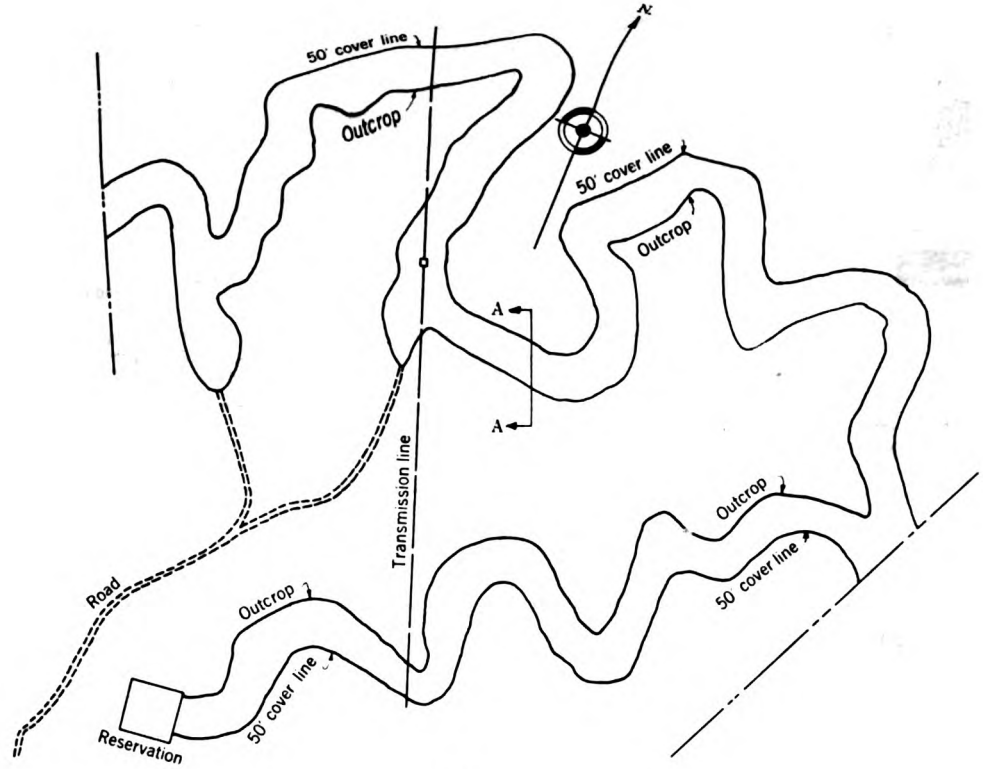

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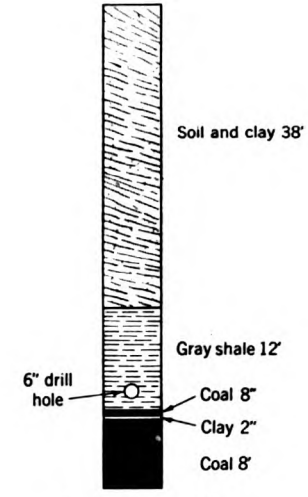
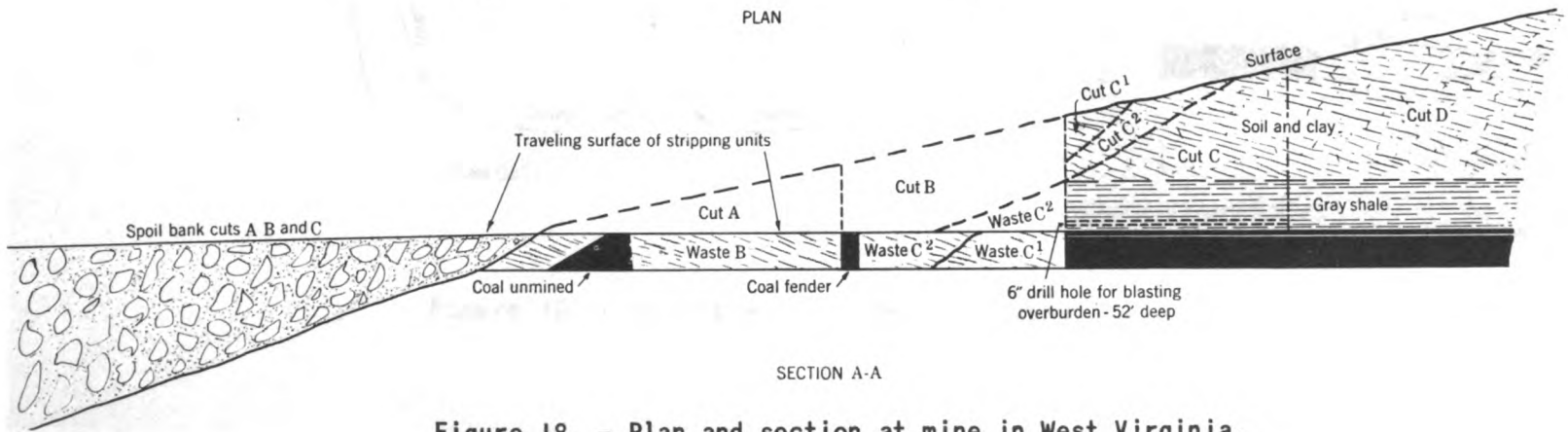


Section of coal bed and overlying strata



SECTION A-A

Figure 18. - Plan and section at mine in West Virginia.

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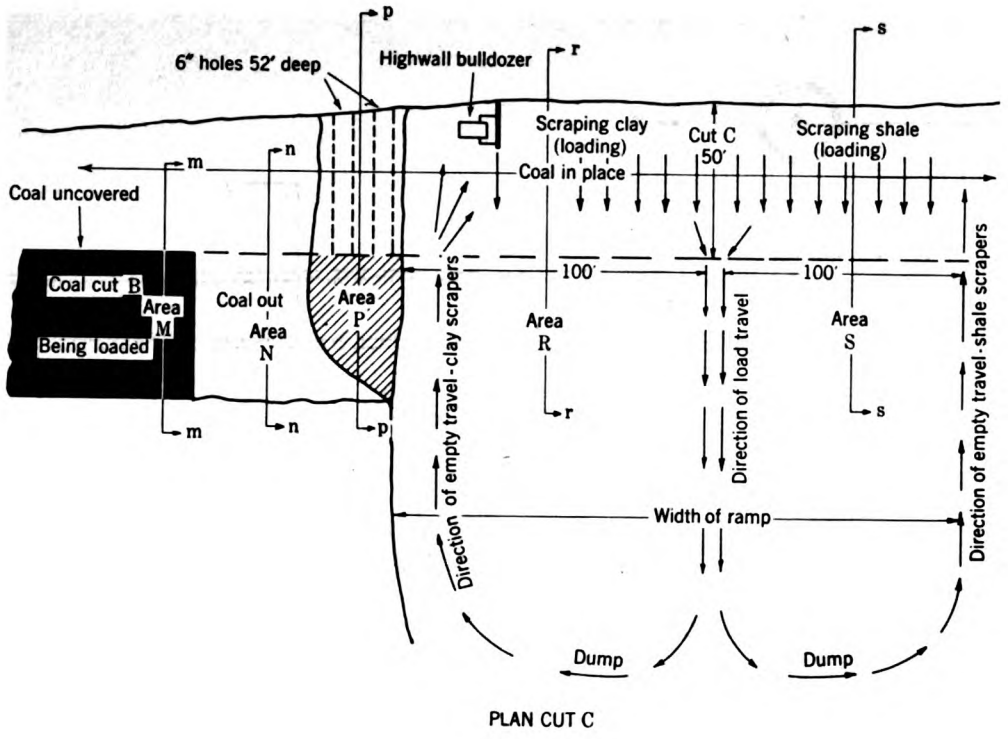

PLAN CUT C

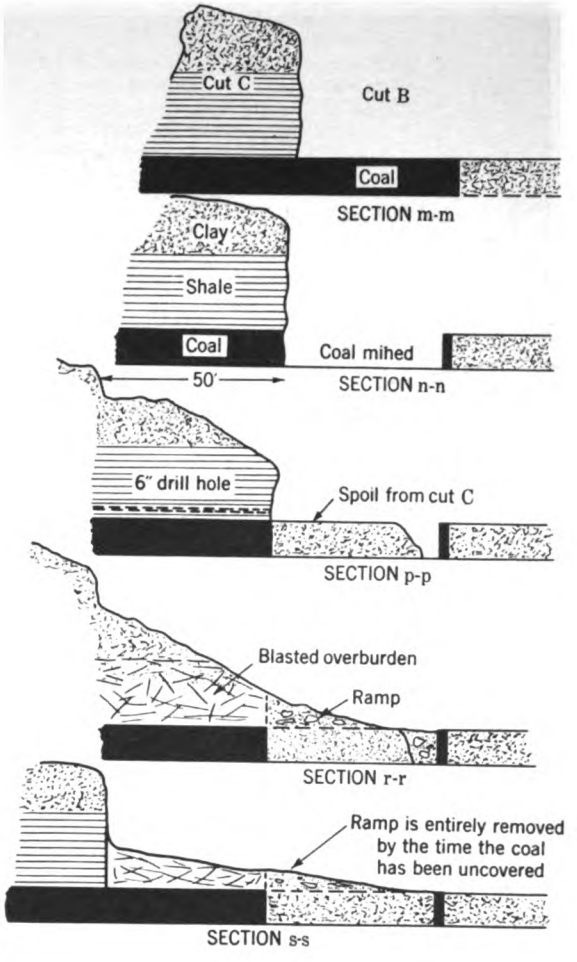

Figure 19. - Mining method, West Virginia.

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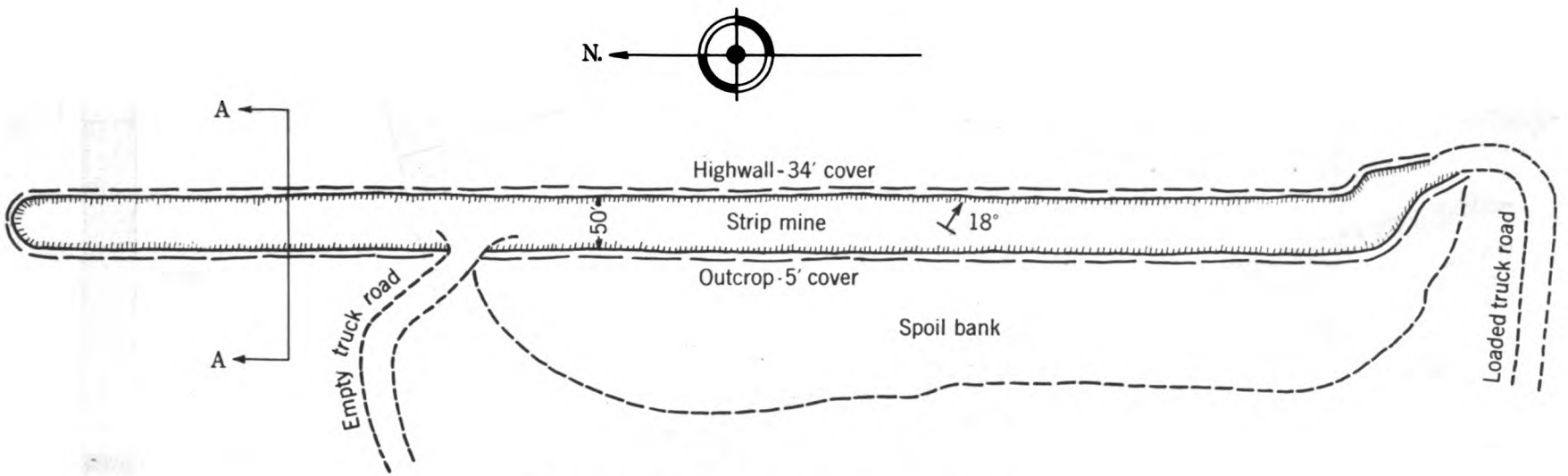

Figure 20. - Plan of mine, Maryland.

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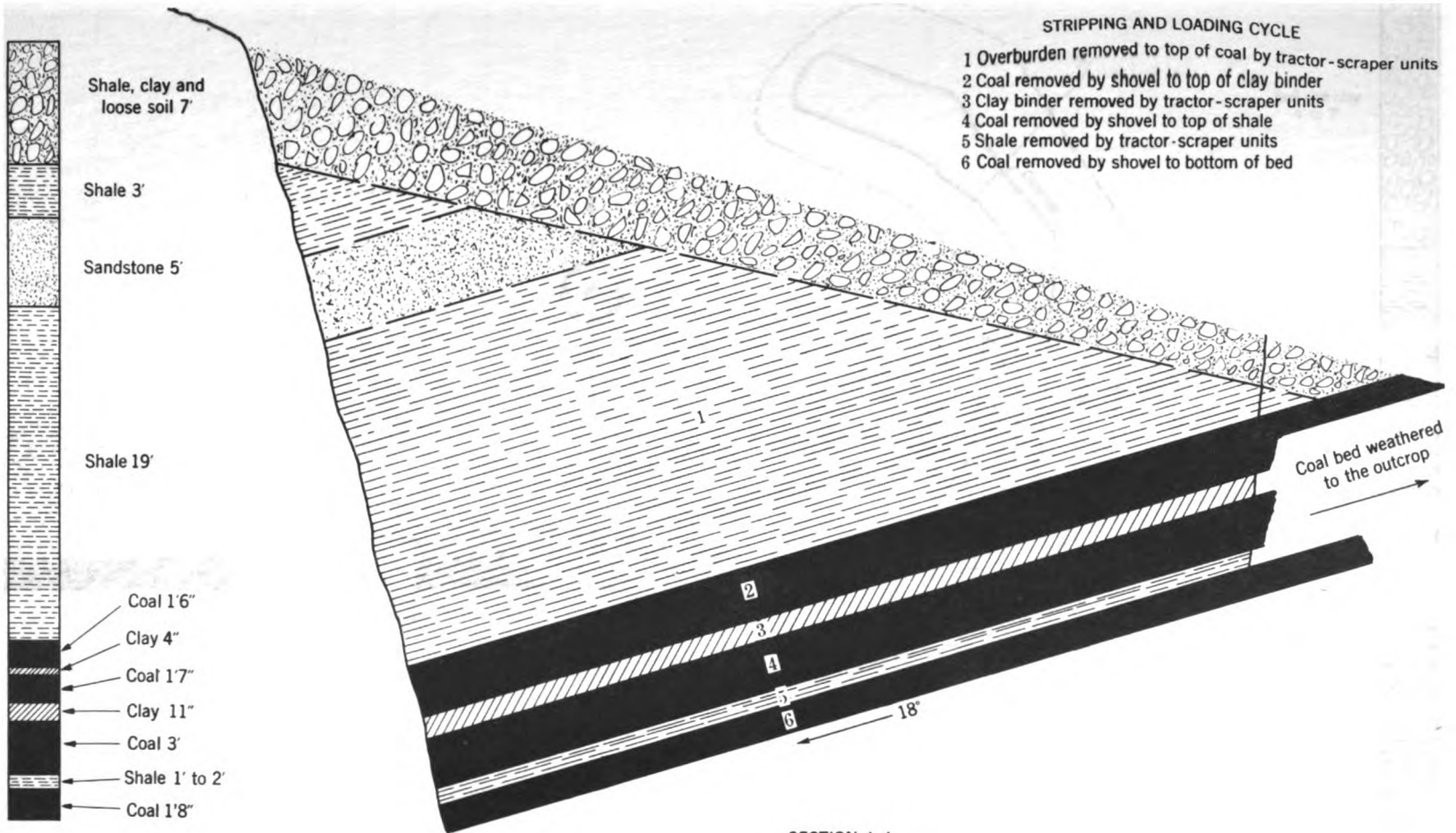
STRIPPING AND LOADING CYCLE
1 Overburden removed to top of coal by tractor-scraper units
2 Coal removed by shovel to top of clay binder
3 Clay binder removed by tractor-scraper units
4 Coal removed by shovel to top of shale
5 Shale removed by tractor-scraper units
6 Coal removed by shovel to bottom of bed
Shale, clay and loose soil 7'
Shale 3'
Sandstone 5'
Shale 19'
Coal 1'6"
Clay 4"
Coal 1'7"
Clay 11"
Coal 3'
Shale 1' to 2'
Coal 1'8"
Coal bed weathered to the outcrop
18"
SECTION A-A

Section of coal bed and overlaying strata mine 3

Figure 21. - Section of mine and mining cycle, Maryland.

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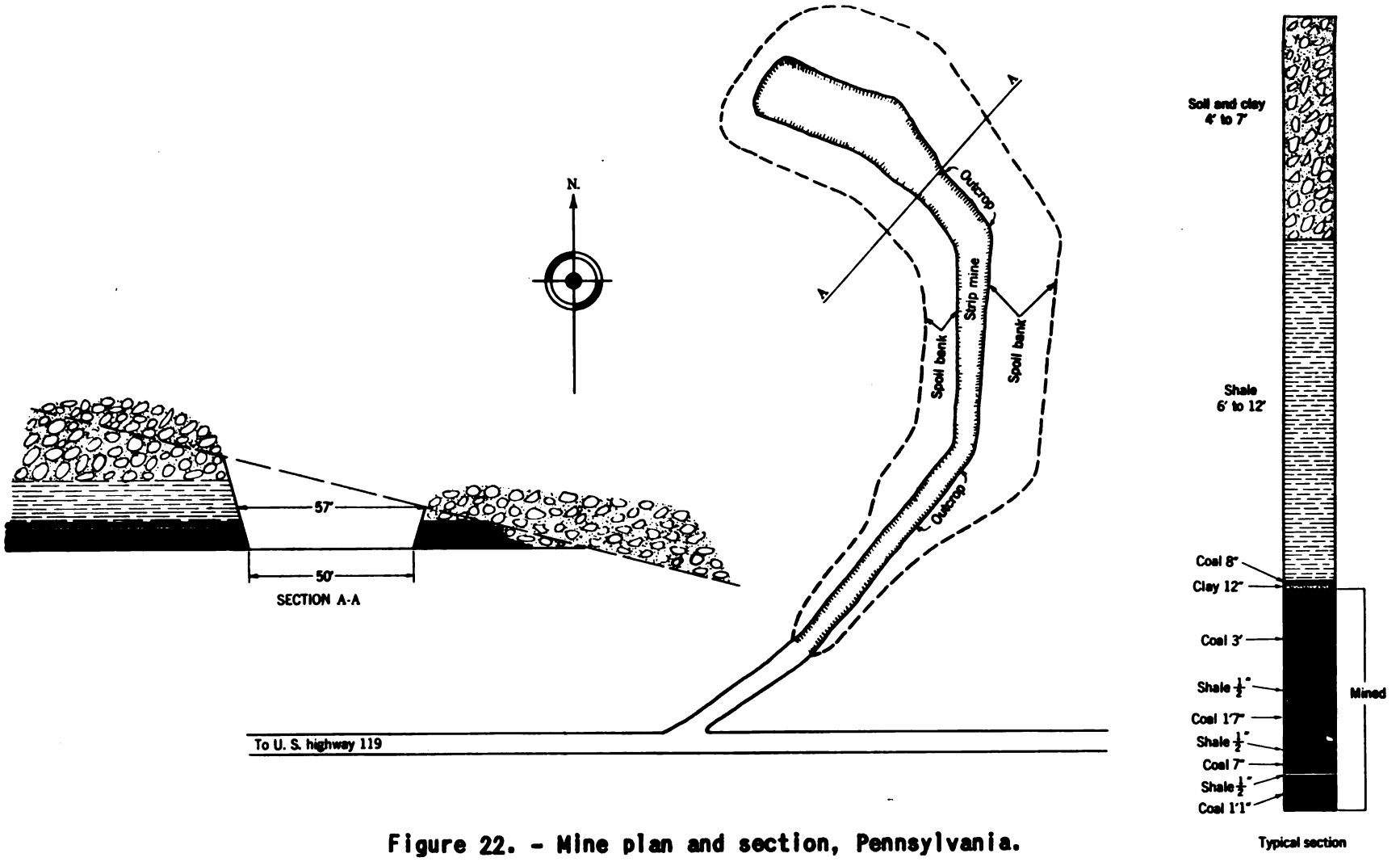

Figure 22. - Mine plan and section, Pennsylvania.

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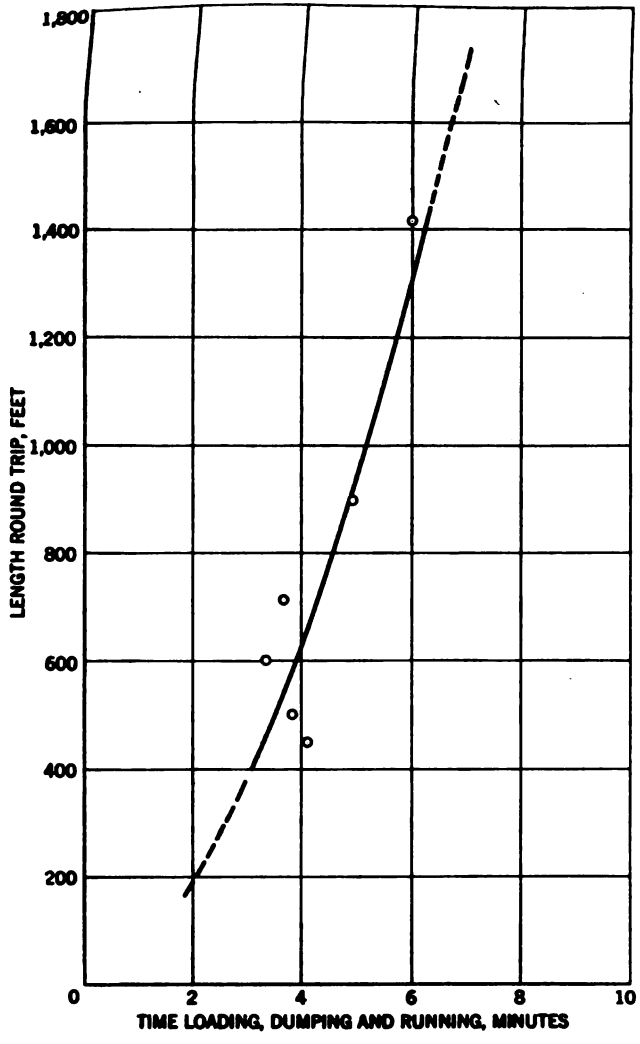

Figure 23. - Relation of average round-trip cycle (feet) to time (minutes) for tractor-scrapers.

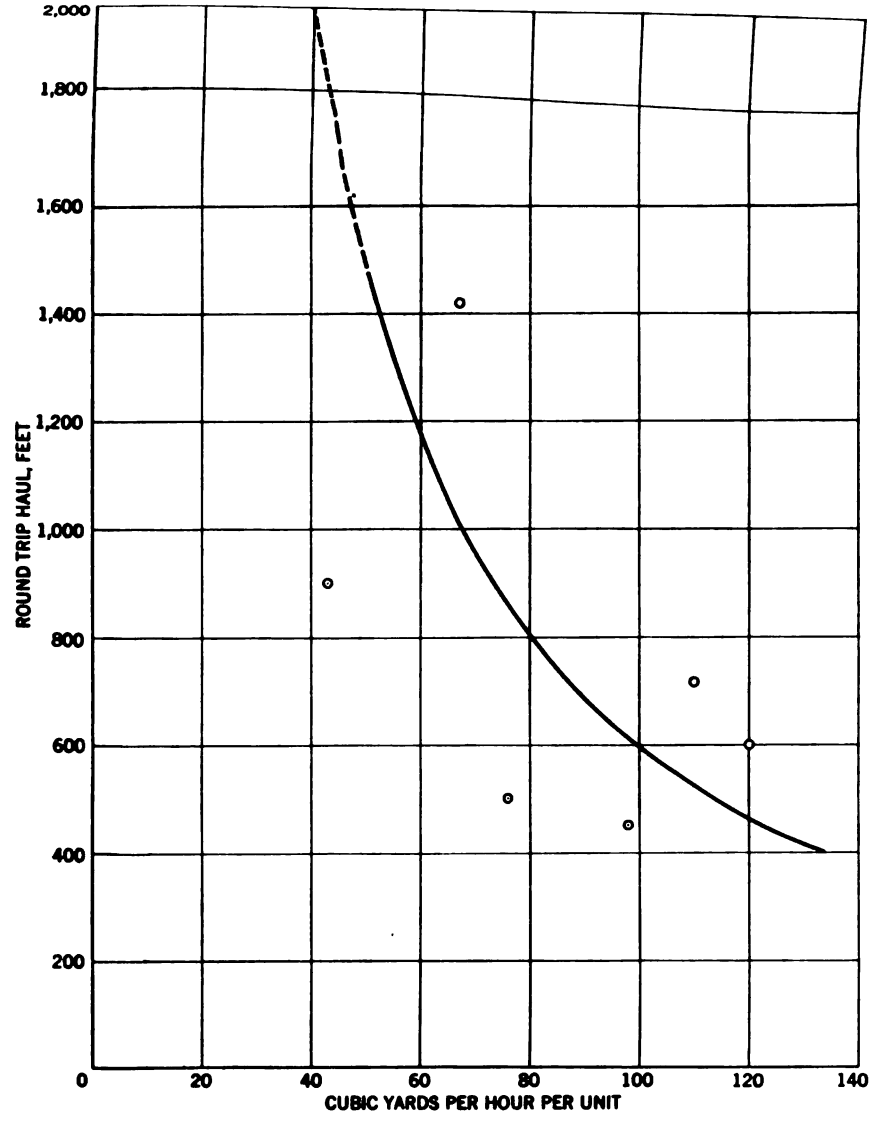

Figure 24. - Relation of cubic yards transported per hour per tractor scraper to round trip (feet).

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At four mines, the overburden is blasted to obtain fragmentation before the material is moved. Horizontal holes 6 inches in diameter are drilled in the shale 2 to 2-1/2 feet above the top of the coal bed. The holes are spaced 12 to 20 feet apart, depending on the thickness and character of the overburden and drilled 45 to 54 feet deep, depending on the width of the cut desired. Holes are charged with 150 to 257 pounds of explosive, which is detonated electrically.

The rated capacity of the scrapers used is 12, 15, and 25 cubic yards, and these units carry an average payload of 8, 10, and 18 cubic yards, respectively. The scrapers are pulled by Diesel-powered tractors and pushed by Diesel-powered pushers or bulldozers. (See figs. 16 and 17.) The average length of round-trip hauls varies considerably, owing principally to the topography of the surface and the amount of spoil to be moved beyond the outcrop. Usually, bulldozers shape and grade the spoil banks away from the strip mine in favor of drainage. Mining methods followed at mines studied in West Virginia, Maryland, and Pennsylvania are shown in figures 18, 19, 20, 21, and 22.

At most of the mines visited, the coal required drilling and blasting. Holes are spaced on 4- to 9-foot centers, depending on the hardness of the coal. The coal is loaded into trucks with small Diesel- or gasoline-powered shovels, which operate on the clay bottom underlying the coal. At three mines, the loaded coal trucks operate on top of the coal bed and at the other mines on the underlying clay. The coal is transported by trucks to tipples, which usually are at railroad loading points. These trucks operate over roads built and maintained by the operators or over State and county highways. The coal is hand-picked at the tipple and usually is loaded into railroad cars as run-of-mine. However, at two mines, the strip-mined coal is mixed with coal from underground mines and passes through the screening plants at these mines.

The following curves were plotted from time studies and other data obtained:

Figure 23. - Relation of average round-trip cycle (feet) to time (minutes) for tractor-scrapers:

> This curve is based upon the weighted average performance of tractor-scraper units at all mines studied and reflects the time required for these units to complete round trips for distances ranging from 200 to 1,800 feet. All tractor-scrapers are operated under similar physical conditions, although they are of different capacities. The grouping of points computed for each mine is near the curve and shows that the size of the scraper has little relation to the time cycle. The rate of increase in time to complete the cycle decreases as the length of round-trip haul increases.

Figure 23 shows the advantage of maintaining short hauls. For example, there is an increase of 35 cubic yards transported

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per hour per tractor-scraper when the round-trip distance is reduced from 600 to 400 feet and only 8 cubic yards per hour when the round trip distance is reduced from 1,200 to 1,000 feet.

The conclusions reached during the investigation are summarized as follows:

1. Light earth-moving equipment is particularly adapted to hillside and hilltop stripping in hilly and mountainous areas because of the mobility of these machines. This type of equipment also has the advantage of being able to move itself to the mine site. Bulldozers and tractor-scrapers build roads to the mine, and these roads are used later in the transportation of the coal produced from the mine and material and supplies to the mine.

2. Tractor-scraper operation should be planned so that the round-trip haul is held to a minimum. The most efficient length of round-trip haul (fig. 24) appears to be 800 feet or less.

3. The length of cut that should be made by tractor-scrapers on the high wall for efficient operation depends on the compactness of the overburden. Scrapers can make deeper cuts in soft than in hard material, and the length of cut should be the shortest distance required to load the scraper to capacity without stalling the pulling or pushing tractors. Therefore, the shortest round-trip haul at any strip mine depends on the length of cut and the distance from the cut to the spoil pile.

4. Tractor-scrapers often are used as auxiliaries to stripping shovels. The overburden beyond the limits of the stripping shovels is moved by tractor-scrapers, and this practice enables the shovels to uncover coal that otherwise might be lost.

5. The law of some States requires restoration of the surface after the coal has been mined by stripping. Bulldozers, tractor-scrapers, and dump wagons are adapted to this work, because spoil piles can be shaped as stripping progresses.

6. Tractor-scrapers do not operate efficiently when digging wet, sticky material and are not suitable for digging rock.

Mining in Belgium and Germany

During the war, as rapidly as the Allied Forces regained European territory, it was necessary to see that the liberated people were supplied with fuel as well as other things. The conditions prevailing in Europe and the methods used to provide as much coal from local sources as possible was of a great deal of interest to the American people. Dr. Louis C. McCabe, formerly a colonel attached to SHAEF, was in charge of coal mining and distribution in Belgium and later in the Allied occupation zone of Germany as well. The activities in Belgium immediately after liberation and the entire Allied zone following the defeat of Germany has been discussed many times, but has appeared

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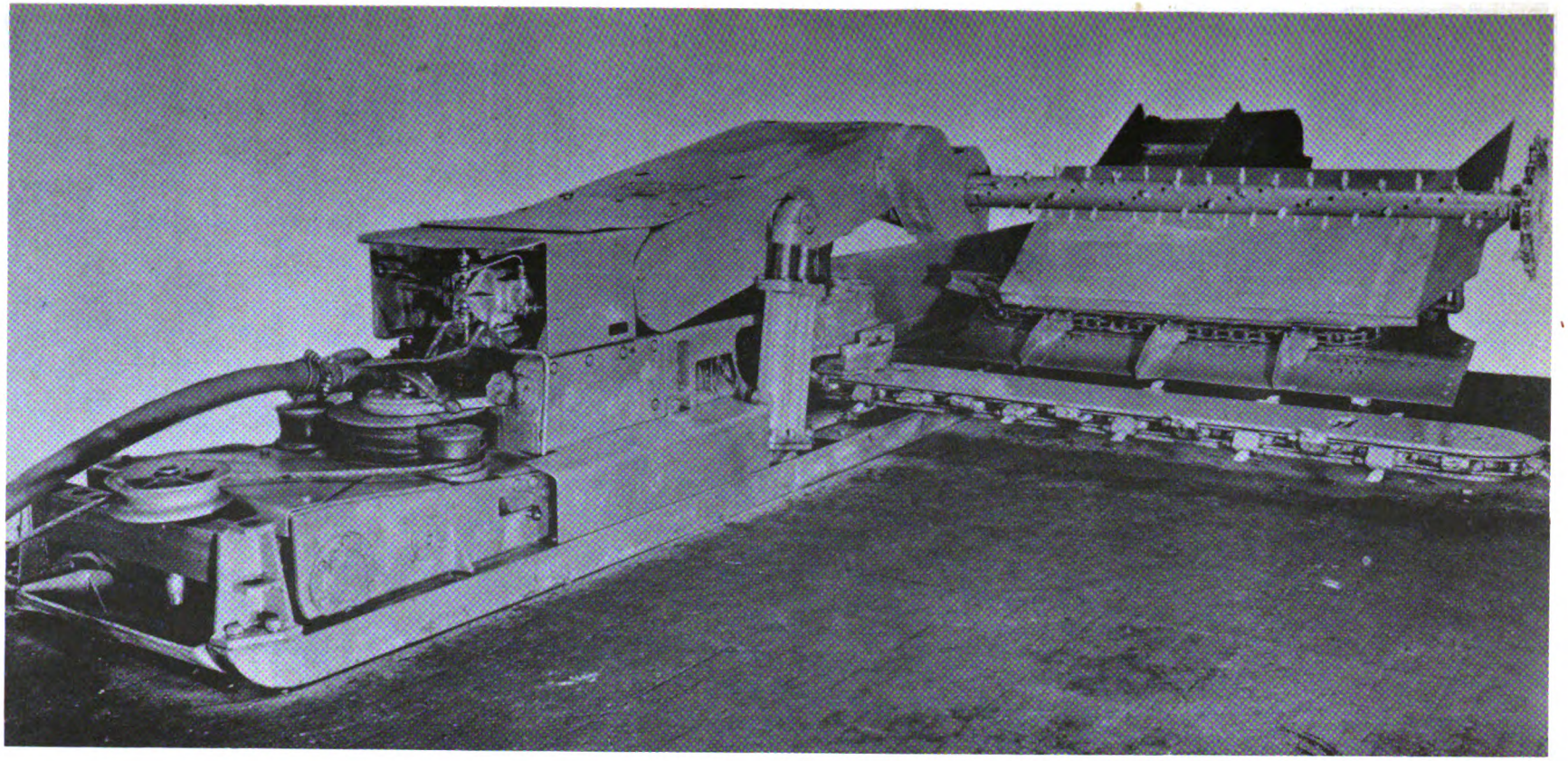

Figure 25. - Eickhoff Schrämlader (coal cutting and loading machine).

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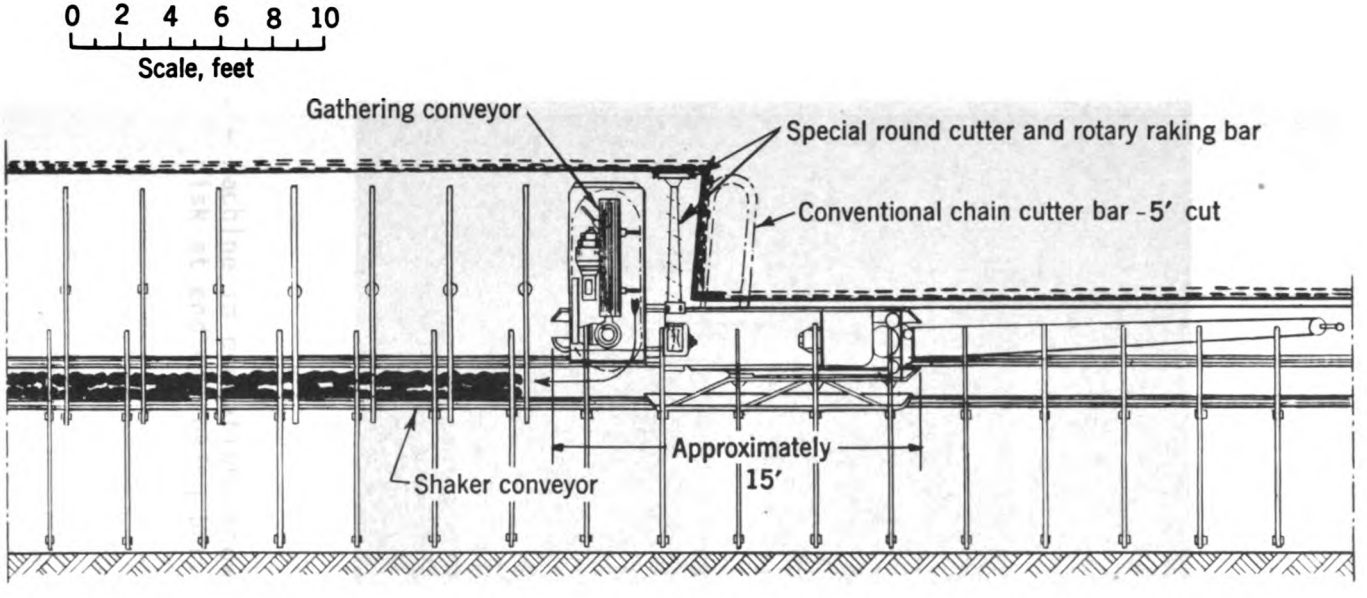

Figure 26. - Eickhoff Schrämlader (coal cutting and loading machine), showing arrangement of gathering conveyor loading directly into shaker conveyor.

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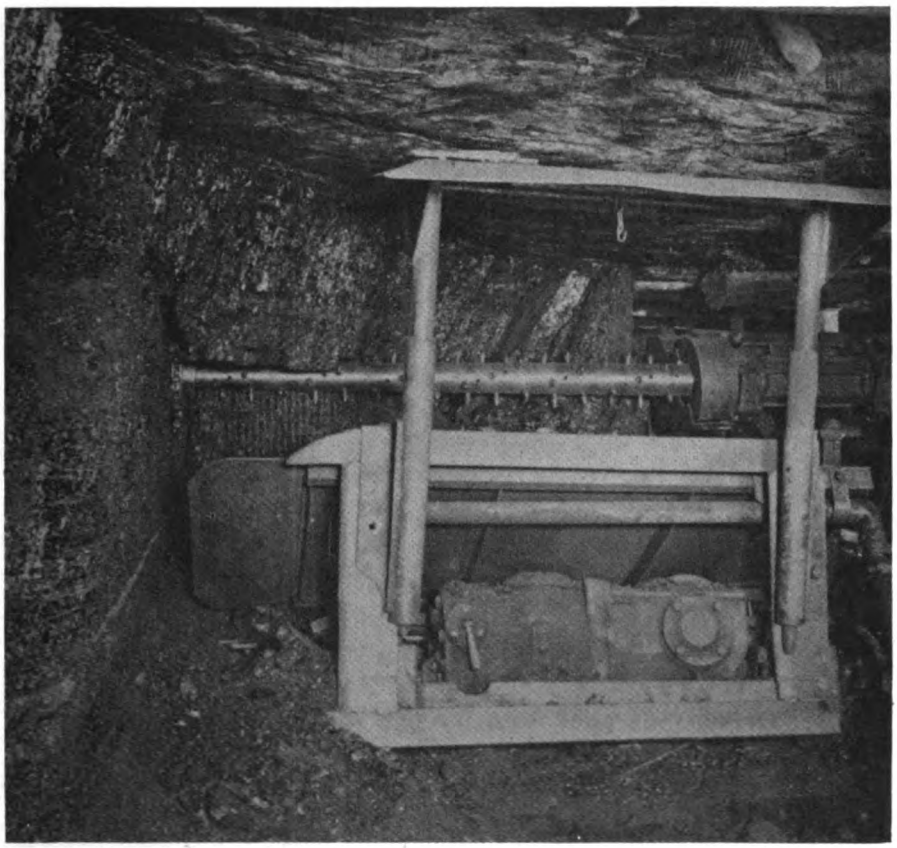

Figure 27. - Machine in operation, showing cut made by disk at end of raking bar.

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in published form only once.17/ Methods used to increase coal production to meet military and essential civilian needs and the allocation of mined coal, together with a summary of the amount of coal produced in Germany over a several-year period, were reported.

Eickhoff Schrämlader

The mining industry in the United States is interested in new types of mining machinery adaptable to mechanized mining in this country. Detailed German drawings showing the design of a longwall coal cutting and loading machine, developed by Eickhoff Maschinenfabrek, Bochum, Germany, and instructions for its operation, were sent to the Bureau of Mines from Germany by the Solid Fuels Section, SHAEF. The development and testing of this machine in Germany are supposed to have taken place during the latter part of 1941 and early 1942. An engineering description of the machine was published.18/

The Eickhoff cutting and loading machine, shown in figure 25, can be used where underground conditions are favorable for longwall mining. It is not suitable for use in room-and-pillar work. The basic principle of the machine is a typical longwall jackknife coal-cutting arm for cutting a bottom kerf approximately 5 feet deep. An auxiliary cutting unit is mounted on top of the chain cutter car. This auxiliary cutting unit consists of a round raking bar mounted in a substantial supporting arm. This arm is hinged and is moved up and down by hydraulic jacks so as to place the raking bar in any position between the chain cutter bar and the top of the coal. This raking bar dislodges the coal, and the broken coal falls onto a horizontal flight conveyor. This conveyor delivers the coal to a longwall shaker conveyor in the room which transports the coal to the loading point on the entry. (See figs. 26 and 27.)

The machine is reported to have been installed and used in the Jacobi coal mine in Germany and produced the following tonnages during 1942:

1942	Tons
August	10,433
September	10,750
October	12,267

No information is available of the man-hours required to operate the machine, but evidently considerable dead work is required to move it from one working face to another prepared longwall face and then to place it in operation.

This type of machine could be manufactured in the United States if the coal-mining industry desires to experiment with it in longwall mining. Some

17/ McCabe, Louis C., Belgian and German Coal Mining During the European Campaign: Proc. Illinois Min. Inst., 1946, pp. 53-63.

18/ Arentzen, Einar M., Description of the Eickhoff Schrämlader, Longwall Coal Cutting and Loading Machine: Bureau of Mines Inf. Circ. 7390, 1946, 5 pp.

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of the cutting elements of the machine might be combined with mechanical loaders now used in this country and result in more flexible operation than the use of the conveyor arrangement of the Eickhoff machine.

Bureau of Mines Scraper-Shaker Loader

A scraper-shaker loader was designed and built by the Bureau to solve the problem of transportation delays in mechanizing development work in thin, steeply pitching anthracite beds. It consists of a standard scraper loading slide, having a hoe-type scoop which discharges into the drive pan of a shaker conveyor. The drive pan is permanently installed as a part of the loading slide and actuated by a standard shaker-conveyor drive permanently mounted upon the truck of the scraper-loading slide. Both scraper and shaker are compressed-air-powered, since there is no electrical distribution system underground in the condition for which it was built. Animal haulage is used exclusively in this mine. By extending the shaker-conveyor trough line, which is suspended from the roof at greater than car height, enough mine cars can be placed before loading begins so that one full face cut can be loaded mechanically without excessive transportation delays attributed to animal haulage.

Preliminary tests of this loading machine have been completed, and its practicability has been proved. Using it a rate of advance double that of single-car scraper loading was shown to be attainable, with a rate of advance six times that obtained by hand methods. The machine has been removed in order to incorporate improvements in design which the experimental work indicated.

Eickhoff Shearing Machine

An Eickhoff shearing machine, model DEK, of German origin was imported for test purposes in productive mining of thin, steeply pitching beds of anthracite. This machine, which weighs only 1,500 pounds complete with caterpillar mount, makes a vertical shear cut 3 inches wide and 5-1/2 feet deep. Driven by a compressed-air motor of 9-horsepower rating, the machine developed 15.3 horsepower under Bureau tests, although 6 horsepower was found to be sufficient for cutting anthracite with it. The caterpillar mount will be replaced by a skid frame for driving 30° slant chutes in a bed pitching 85° under a new plan being developed for mechanizing heavy-pitch work.

Korfmann Universal Shearing Machine, Model SK 20

A Korfmann universal shearing machine, model SK 20, was imported from Germany to be tested in the anthracite region of Pennsylvania. This machine, weighing approximately 2 tons, is driven by a compressed-air motor rated at 20 horsepower. Within the practical operating ranges, Bureau tests showed a maximum output of 16.1 horsepower, which is more than twice the power required to cut anthracite. The universal feature permits unlimited multi-directional cutting by revolving the machine head, through 360°, to the position desired. This machine is mounted on a car-wheel truck and will be remounted on caterpillars for driving a trackless gangway 1,600 feet in length.

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German Coal Planer

The rigid-blade coal planer[19] was fully developed in wartime Germany; but in that form can only be used for mining friable coal beds or on long-wall faces where closely controlled roof pressure is used to break up the solid coal face sufficiently in advance of the mining. The vibrating-blade planer, intended for use in harder coals, had not been fully developed by the war's end. The Bureau has undertaken the design, manufacture, and development of such a vibrating-blade planer for tests in anthracite mines. Investigations showed pneumatic hammers to be more suitable than mechanical or electrical devices for the vibrating action, and four special hammers for use in the Bureau's planer are now in the process of manufacture. Intended for trial in thin, flat, virgin beds of the Wyoming region, successful application of the planer principle to anthracite will open a way to mine the 50-foot heavily pitching beds of the Southern field mechanically. It was found that the best conventional mining practice in the German Ruhr district was improved more than threefold by introduction of the coal planer.

Anthracite Flood Prevention

The flooding of active and abandoned anthracite mines in certain areas of Pennsylvania is a constant menace to life and property, threatening to curtail the life of the anthracite industry and to decrease the present production of anthracite. The problem of flooding was investigated with consideration given to three main sources, which are: (1) Main- and side-stream leakage, (2) general surface leakage, and (3) barrier-pillar seapage. Billions of gallons of impounded water threatening active workings have been located and shown on maps.[20] The general topography in the anthracite region is such that water from the steep mountains tends to collect in almost every man-made opening, whether it is a stripping or underground mining operation. Some measures have already been taken in the region to divert water. However, there remain many properties separated from water pools by barrier pillars which probably are not strong enough to maintain the head of water against them. In other instances, particularly in the Susquehanna Valley in the Northern field, the water channels underneath present river beds are so close to the coal measures that unless special precautions are taken the water may break through, causing an enormous loss of coal. The details of the solution of the flooding problem cannot be postulated until a comprehensive study of the entire mine water problem has been made.

Acid Mine Water from Anthracite Mines

The mine-drainage systems in the anthracite region handle over 200 billion gallons of water annually, of which 150 billion gallons is pumped to the surface. Much of this water is being utilized for breaker use, dust-control

19/ Buch, John W., Design and Operation of the Coal Planer, Ruhr District, Germany: Bureau of Mines Inf. Circ. 7377, 1946, 20 pp.

20/ Ash, S. H., and Westfield, James, Flood-Prevention Projects at Pennsylvania Anthracite Mines: Bureau of Mines Rept. of Investigations 3868, 1946, 25 pp.

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installations, and hydraulic back filling, as well as to combat mine fires and aid in the transportation of anthracite in gently dipping places where sheet iron is used.21/ It is estimated that 1,100 gallons of mine water is utilized in preparing each ton of anthracite mined.

Much of the mine water used is highly acid, and, unless treated to reduce the acidity, the replacement and labor costs required to maintain coal preparation at desired capacity are exorbitant.

The treatment of mine water for washery use by lime reduces maintenance costs considerably.

The first installation for lime treatment of mine water used for preparing anthracite was put into operation in 1932. The four types of installations used are the screw feeder, the vibrating feeder, the disk feeder, and tanks in which lime and water are mixed.

At present, hydrated lime is the only kind of lime used in the anthracite region to treat acid water.

The advantages of treating acid mine water with lime are shown by comparing the cost of breaker materials before and after treatment and by the favorable statements of individuals in charge of the lime-treatment installations.

Anthracite-Mine Dust Problems

Studies have been conducted by the Bureau of Mines in the anthracite region of Pennsylvania, in cooperation with several large mining companies, to develop methods for eliminating or reducing the dissemination of dust into the air by various mining practices. No single method of control is applicable to all dusty operations, and several methods have been practiced with considerable success. Preventing the dissemination of dust by undercutting machines, pneumatic drills, blasting, loading, and transportation of coal is discussed. The methods presented are practical and were successful in markedly reducing the dust concentration in the breathing zone of the worker. Such dust-control procedures not only reduce possibility of injury to health but also increase the efficiency and morale of the worker.22/

Back Filling in Anthracite Mines

Mine subsidence causes a great deal of damage to surface ground and structures and also endangers mine workings. The Pennsylvania anthracite region

21/ Johnson, Leland H., Treatment of Acid Mine Water for Breaker Use in the Anthracite Region of Pennsylvania: Bureau of Mines Inf. Circ. 7382, 1947, 14 pp.

22/ Johnson, Leland H., Dust Problems in the Mines of the Pennsylvania Anthracite Region: Bureau of Mines Tech. Paper 704, 1947, 34 pp.

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has been studied.[23] Possibilities of using several methods of back filling more extensively are presented.

Diesel Exhaust Gases

The effect on quality of tunnel air resulting from the operation of Diesel locomotives is discussed in a report[24] that describes the use of this type of equipment in construction of some of the tunnels of the Delaware Aqueduct in New York State. The selection of Diesel-powered equipment for these operations was based upon apparent advantages in initial cost and in performance on contracts where haulage conditions were adverse.

The first locomotive built for this purpose was factory-tested by the Bureau of Mines to obtain an estimate of the rate of ventilation that would be required when the locomotive was placed in actual service. After this locomotive and others were in use in the tunnels, periodic examination of the tunnel air showed that, under normal operating conditions, the carbon dioxide, carbon monoxide, and oxides of nitrogen content of the air did not exceed limits generally considered permissible in the air of working places, nor was the oxygen content depleted to any significant extent.

These field studies on the use of Diesel engines underground and laboratory experiments showed that the odorous and irritating properties of Diesel exhaust gases are related to the concentration of aldehydes in these gases. In many applications of Diesel engines, it would be desirable to remove aldehydes and eliminate or minimize the odor and irritation. Attempts to do this by scrubbing with water in full-scale laboratory tests showed that only partial removal of aldehydes is obtained under practical operating conditions. Accordingly, the development of an economical method for complete removal of aldehydes from Diesel exhaust gas was undertaken.

A survey of the literature disclosed that the reaction $Na_2SO_3 + HCHO + H_2O \rightarrow NaOH + CH_2(NaSO_3)OH$ might be utilized in removing aldehydes from Diesel exhaust gas. The applicability of this reaction to the problem was studied in a series of bench-scale experiments in which air containing approximately 50 ppm of formaldehyde was scrubbed with aqueous solutions of sodium sulfite. These tests showed that substantially complete removal of aldehydes was obtained for comparatively long periods at temperatures up to 140° F. and with solutions containing 2 to 5 percent sodium sulfite. These tests showed also that the removal of formaldehyde decreased markedly when most of the sodium sulfite had been oxidized to sodium sulfate. Since oxidation will occur under practical exhaust-gas scrubbing conditions, it is necessary to inhibit oxidation of sodium sulfite to obtain effective aldehyde

23/ Ash, S. H., and Westfield, James, Back-Filling Problem in the Anthracite Region as It Relates to Conservation of Anthracite and Prevention of Subsidence: Bureau of Mines Inf. Circ. 7342, 1946, 18 pp.

24/ Berger, L. B., Elliott, M. A., Holtz, J. C., and Schrenk, H. H., Diesel Engines Underground. VI. Use of Diesel Locomotives in Construction of the Delaware Aqueduct; Effect of Exhaust Gases Upon Quality of Tunnel Air: Bureau of Mines Rept. of Investigations 4032, 1947, 26 pp.

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removal for extented periods. Previous work outside the Bureau of Mines[25] had shown that hydroquinone was the most effective inhibitor for the oxidation of sodium sulfite. Under the conditions of the bench-scale tests at 160° F., the addition of 0.3 percent hydroquinone to a 5 percent sodium sulfite solution significantly retarded oxidation. For example, at the end of 5 hours the concentration of sodium sulfite was 3.1 percent without inhibitor and 3.9 percent with inhibitor.

A series of full-scale scrubbing tests was made with a scrubbing solution containing 10 percent sodium sulfite and different concentrations of inhibitor. The scrubber used was very simple and consisted essentially of a perforated pipe submerged in the scrubbing solution. The temperature of the scrubbing solution was about 130° F., and the throughput of exhaust gas at NTP was approximately 3,000 cu. ft./hr. The concentration of aldehydes in the inlet gases ranged from 6 to 60 ppm. The results of these tests are shown in figure 28. It will be observed that with no inhibitor the removal of aldehydes decreased markedly after about 2 hours, whereas, with 0.5 percent hydroquinone inhibitor, removal was satisfactory for 15 hours. From these results, it is apparent that a large fraction of aldehydes can be removed from Diesel exhaust gas for extended periods. In the practical application of this method, the concentration of sodium sulfite and inhibitor will depend on the conditions of operation. The cost of the components of the scrubbing solution is nominal, so that the solution may be discarded when it is spent.

Combustion Performance of Fuels in Diesel Engines

In preparation for work on the performance of different fuels in Diesel engines, a series of tests was made in the CFR Diesel engine to determine the combustion performance of this unit and to assess its utility in combustion studies. The results of some of these tests made at a compression ratio of 15:1 and with injection of a commercial Diesel fuel having a 55 cetane number are shown in figure 29. This figure clearly shows the existence of two types of smoke in the exhaust from Diesel engines: (1) Unburned or partly burned fuel (predominating at low fuel:air ratios); and (2) unburned carbon (predominating at high fuel:air ratios). At the low fuel:air ratios very little fuel is burned, since figure 29 shows very little carbon dioxide and considerable particulate material in the exhaust gas. As the fuel:air ratio increases a point is finally reached (0.02 pound of fuel per pound of air) at which substantially all fuel is burned. The region between 0.01 and 0.02 fuel:air ratio is somewhat analogous to the lower limit of inflammability. At higher fuel:air ratios the tendency to form locally overrich regions increases, and smoke consisting chiefly of unburned carbon increases. The results in figure 29, when compared with similar results for commercial Diesel engines, show that the combustion performance of the CFR Diesel engine is inferior. In spite of this, the CFR engine may have certain advantages in the study of the combustion performance of fuels, because it accentuates faulty performance.

25/ Kia-Khwe, J., and Alyea, H. N., A Comparison of Organic Inhibitors in Chain Reactions: Jour. Am. Chem. Soc., vol. 55, 1933, pp. 575-588.

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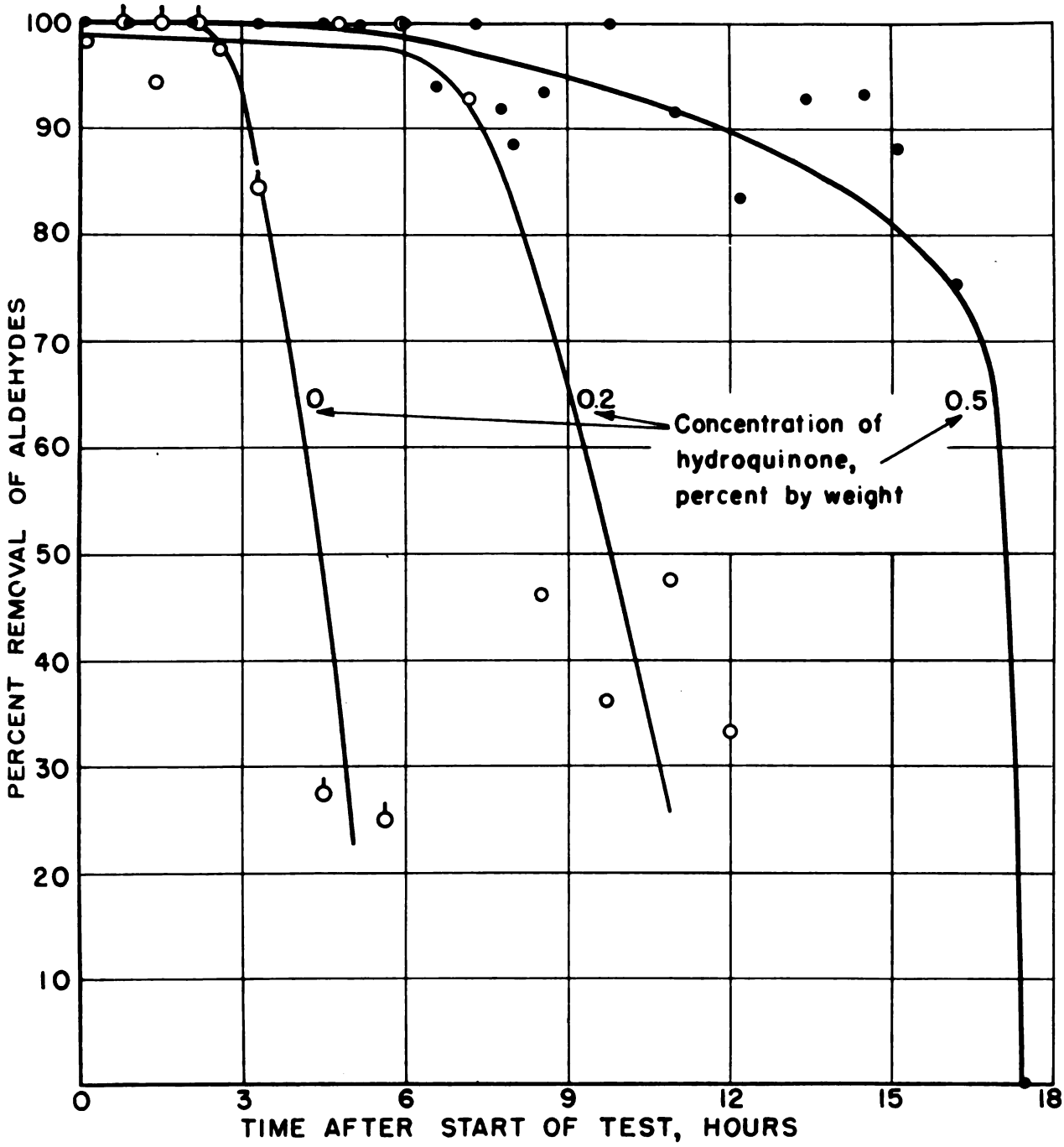

Figure 28. - Effect of inhibitor on removal of aldehydes from Diesel exhaust gas by 10-percent sodium sulfide solutions. (Temperature of scrubber, 133° F.)

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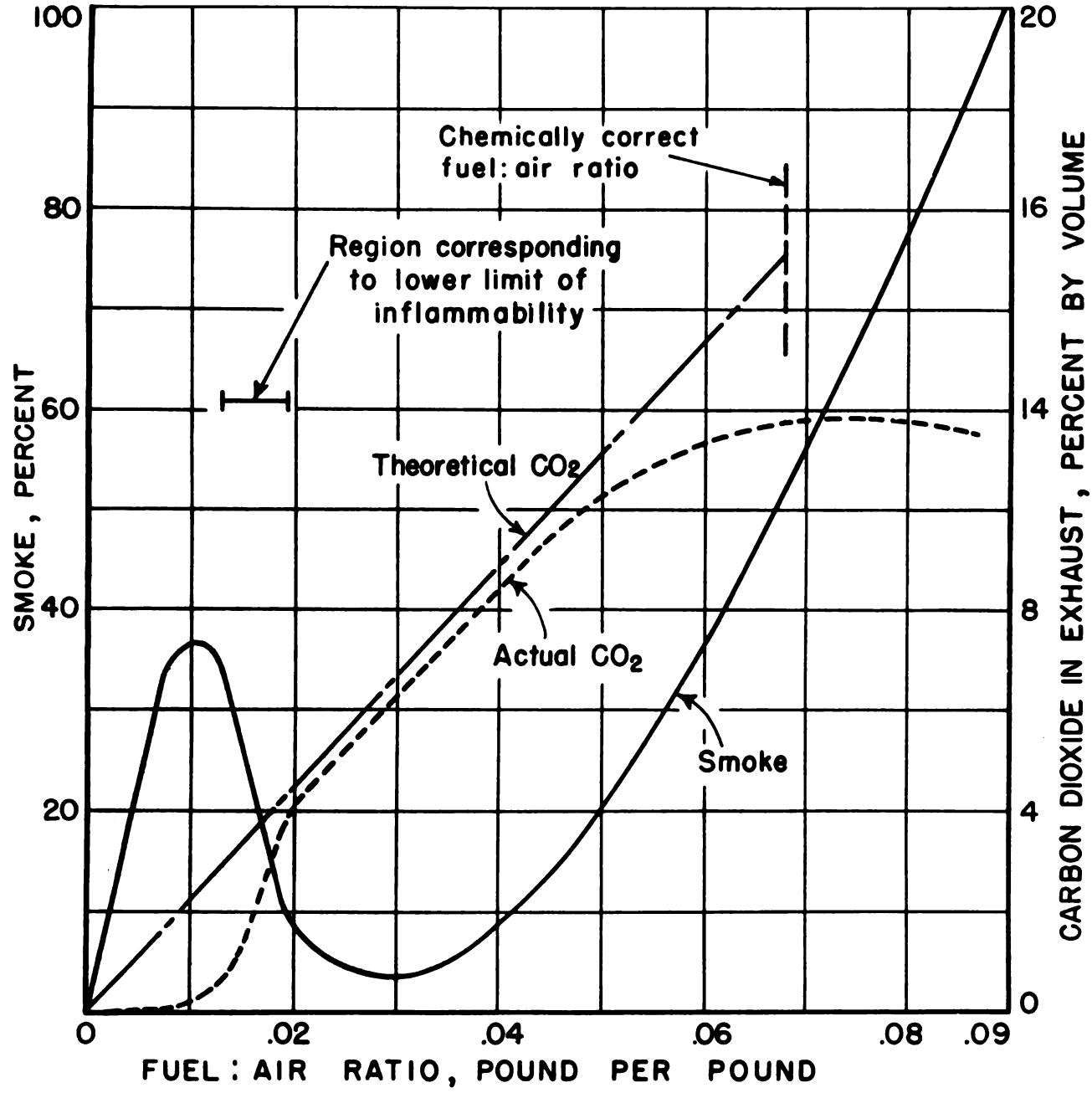

Figure 29. - Effect of fuel:air ratio on carbon dioxide and smoke in exhaust gases from a CFR Diesel engine operated at a compression ratio of 15:1.

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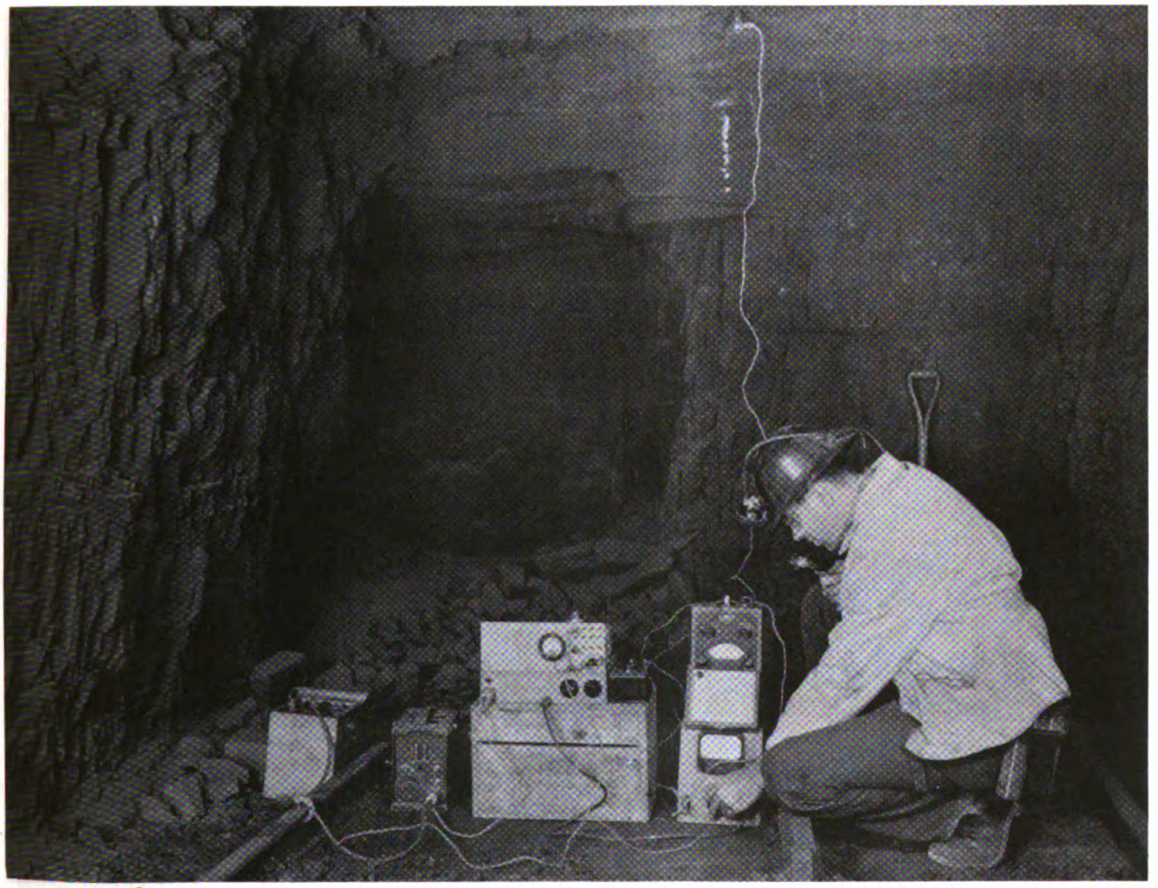

Figure 30. - Experimental apparatus used in developing equipment for 2-way conversation between underground and surface stations.

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Electrical Equipment for Mines

From its inception, the Bureau of Mines has investigated electrical devices and machines available to mines for the purpose of encouraging the development and use of equipment that would help to prevent fires, explosions, and shock in coal mines. The investigation of such equipment is conducted at the Central (Pittsburgh, Pa.) Experiment Station by the Electrical-Mechanical Section of the Safety Division in accordance with published regulations termed "schedules." Equipment that passes the prescribed tests and inspections is formally approved and then is designated "permissible" equipment, that is, permissible for use in gassy coal mines. The Federal Mine Safety Code for Bituminous-Coal and Lignite Mines of the United States, effective July 24, 1946, gave permissible equipment substantial recognition as a safety measure, and its use was prescribed under definite conditions. After permissible equipment has been installed in a mine, it is highly important that it be carefully maintained; otherwise, unsafe conditions might develop.26/

Under the eight schedules now in effect, a manufacturer can have nearly every type of equipment used at or near the face of active mine workings investigated for its liability to ignite gas or coal dust or a combination of these. During the fiscal year 1947, 26 approvals were issued. These included 9 loading machines, 7 conveyors, 3 distribution boxes, 2 cutting machines, 1 storage-battery locomotive, 1 mining-machine truck, 1 mine tractor, 1 blower-fan unit, and 1 sound-powered telephone.

Requirements for Multiple-Shot Blasting Units

Although there has been a need for permissible multiple-shot blasting units, only one 10-shot generator unit has been approved to date. This, however, was withdrawn from sale when it proved unsatisfactory in service. To encourage the development and manufacture of satisfactory designs, the permissibility requirements were broadened after a study of experimental models was completed.27/

Mine-Communication Apparatus

Emergency mine-communication apparatus, for use in rescuing trapped man, was further developed and tested under varying conditions in both anthracite and bituminous-coal mines. Figure 30 shows an arrangement of apparatus underground for two-way conversation with a station on the surface. Preliminary experiments indicate that, in addition to its primary objective of emergency service, there is also the definite possibility of enlarging the scope of this apparatus to include daily utility, such as communication from moving cages in shafts and from man-cars on slopes to hoisting engineers, thus contributing to safety as well as efficiency of transportation.

26/ Gleim, E. J., Care and Maintenance of Permissible Electrical Mine Equipment: Trans. 34th Nat. Safety Cong., vol. 1, pt. 1, 1947, pp. 285-289.

27/ Bureau of Mines, Schedule 16B, Multiple-Shot Blasting Units: Approved April 16, 1947, 5 pp. Duplicated from Federal Register, vol. 12, No. 82, Apr. 25, 1947, pp. 2650-2651.

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Toxic Mine Atmospheres

In connection with requirements of the Federal Coal Mine Inspection Act, approximately 16,000 gas and dust samples were analyzed, which required more than 110,000 individual determinations. Information was presented on methods for the determination and protection against toxic gases.[28] The significance of Bureau of Mines approval in selection of respiratory protective devices was discussed with emphasis on the selection of the proper device and necessity for proper maintenance.[29]

Relationship of Safety and Efficiency in Coal Mining

Accident records have an important bearing on determination of the most effective methods for preventing accidents at coal mines and hence on the cost and rate of production. Analysis and interpretation of statistical data reveal the prevalent types of accidents and their causes, either on a national or a local basis, and provide information necessary for accident-prevention work.[30] Data are given for the number of fatalities in different branches of the mineral industry, and trends of fatality rates are discussed. There has been a definite downward trend in the fatal-injury rate for the period 1931-46 and a very large decrease in fatal injuries for the past 5 years. The rate for 1946 reached an all-time low.

Gaseous Explosions and Use of Explosives

Increase in Charge Limit of Permissible Explosives

Tests to determine the factors that affect the ignition of gas and dust in a coal mine, using larger weights of explosives, were continued during the year.

The principal findings of this year's investigations[31] are summarized as follows.

One hundred and forty tests were made with blown-out, blown-through, and creviced-type shots.

28/ Schrenk, H. H., Methods for Detection and Protection against Toxic Gases: Trans. 21st Ann. Conference and Exhibit, Western Pennsylvania Safety Council and American Society of Safety Engineers, Western Pennsylvania Chapter, Apr. 23-25, 1946, pp. 38-42.

29/ Pearce, S. J., and Schrenk, H. H., Significance of Bureau of Mines Approval in Selection of Respiratory Devices: Safety Eng., vol. 93, No. 1, January 1947, pp. 54-59, inclusive.

30/ Ash, S. H., Safety in Mining: Min. Cong. Jour., February 1947, pp. 35-38.

31/ Hartmann, Irving, and Greenwald, H. P., Tests of Permissible Explosives in the Experimental Coal Mine: Proc. Coal Mine Inst. America, 1946, pp. 68-84.

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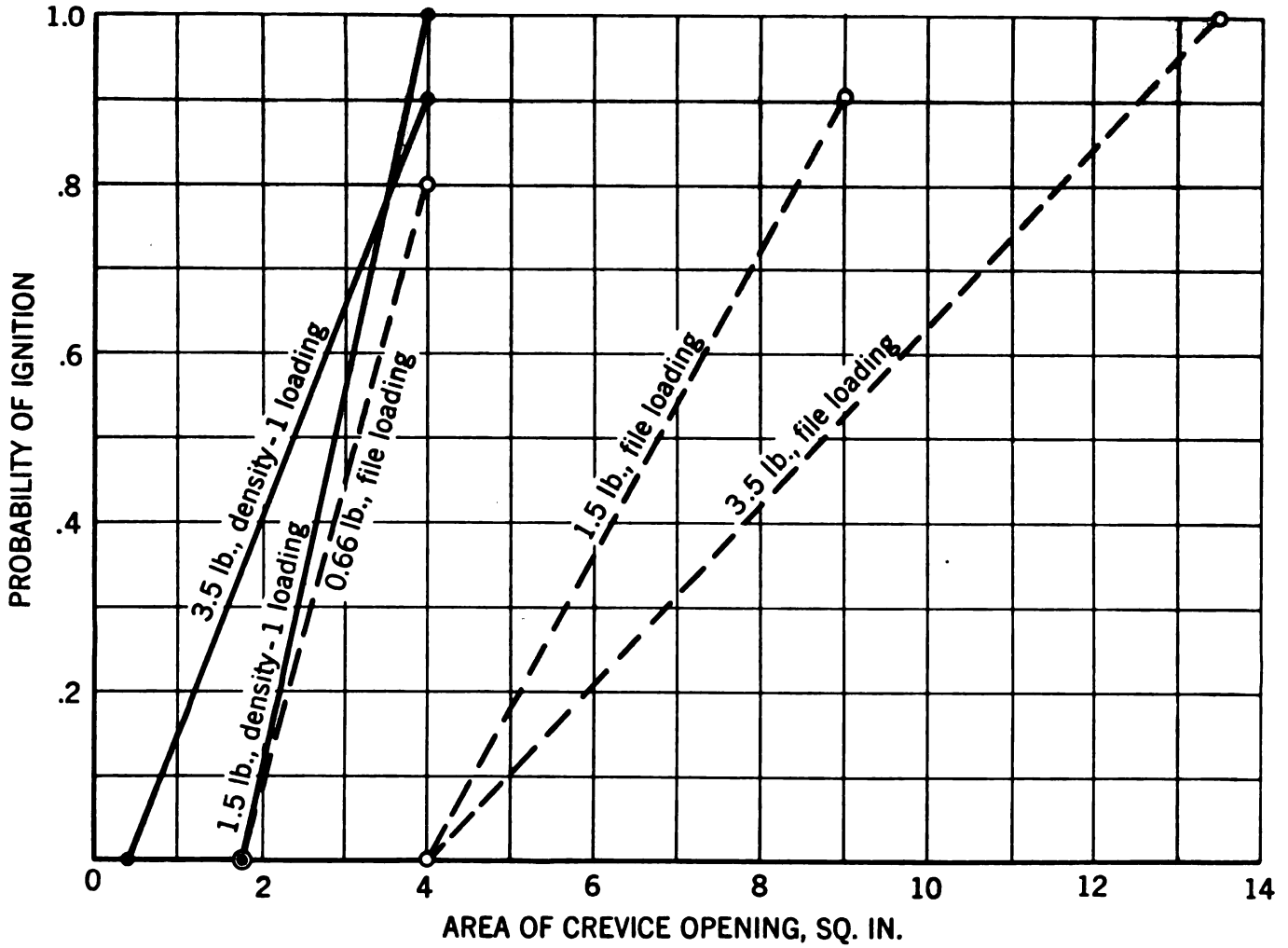

Figure 31. - Effect of weight of explosive and area of crevice opening on probability of gas ignition.

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In previous blown-through tests the charge weights ranged from 3.00 to 8.00 pounds. It has been found that, in general, the ignition hazard increases as the charge weight increases. Recent work has shown that in some cases this relationship may not hold. To be certain that smaller weights of explosive would not be more hazardous in blown-through tests, additional work was done this year with weights of 0.50 and 1.50 pounds of the gelatinous explosive, both file-loaded and at density-1 loading, with a 3-inch burden between the shot hole and the free face. No ignitions were obtained in these tests.

In the creviced-type shots, the results showed that conditions governing ignition of gas are more complicated than in blown-out or blown-through shots. The general results are:

1. With creviced shots, file loading is, in general, safer than density-1 loading, but the difference is not as great as in blown-out shots and under some conditions is reversed.

2. Differences in results were found when the crevice opening was opposite, respectively, the front, center, and rear of the explosive, but these differences are no greater than might occur in duplicate tests.

3. It was immaterial whether the crevice opening was circular or rectangular; the cross-sectional area of the opening was the important factor, provided that the vertical dimension of the crevice did not exceed the diameter of the shot hole. In general, the probability of ignition increased with increased area of crevice. If the crevice was cut so that its height was greater than the diameter of the shot hole, the relationship between the area of the crevice and ignition probability no longer held. The ignition hazard of a long, narrow, vertical crevice is less than that of a long, horizontal crevice.

4. Sheathead explosives were found to be safer than unsheathed explosives, but the gain in safety caused by sheathing was less in creviced shots than in blown-out shots.

5. Weight of explosive was an important factor in an unexpected fashion, as will be indicated below.

The data obtained with unsheathed explosives of varying weights are summarized in the following table. The relation of the groups is shown more clearly by figure 31, on which the area of the crevice opening is plotted along the abscissa and probability of ignition along the ordinate. There are solid lines for two weights of explosive loaded density-1 and dashed lines for three weights of explosive file-loaded.

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Summary of data on weight of explosive and size of crevice opening

Weight of explosive, lb.	Area of crevice opening, sq. in.	Result	Ignition probability
		A. Density-1 loading	
1.50	1.8	0 ignition in 5 tests	0.0
1.50	4.0	5 ignitions in 5 tests	1.0
3.50	.4	0 ignition in 5 tests	.0
3.50	1.8	10 ignitions in 35 tests	.3
3.50	4.0	23 ignitions in 25 tests	.9
		B. File loading	
0.66	1.8	0 ignition in 5 tests	0.0
.66	4.0	4 ignitions in 5 tests	.8
.66	9.0	5 ignitions in 5 tests	1.0
1.50	4.0	0 ignition in 5 tests	.0
1.50	9.0	9 ignitions in 10 tests	.9
3.50	4.0	0 ignition in 10 tests	.0
3.50	9.0	10 ignitions in 35 tests	.5
3.50	13.5	5 ignitions in 5 tests	1.0

The evidence is somewhat surprising. For a crevice having an area of 1.8 square inches, the probability of ignition from 1.50 pounds of explosive loaded density-1 is not greater than from two-thirds of a pound file-loaded. With file loading, the probability of ignition was found to decrease as the quantity of explosive increased. The hazards arising from creviced shots are more severe than those accompanying blown-out or blown-through shots.

Permissibility Tests

Chemical and physical tests of explosives are made to determine their characteristics so that they can be used safely in gassy and dusty coal mines. The conditions and requirements governing the making of these tests are set forth in Schedule 1F.32/

Nine samples of explosives were submitted to determine their safety for use in gassy and dusty coal mines. Seven of these passed the required tests, and two others were withdrawn and not tested. No blasting devices were submitted for tests during the fiscal year.

On June 30, 1947, the permissible list contained the names of 186 explosives and 9 blasting devices as compared with 180 explosives and 9 blasting devices on the list as of June 30, 1945. One of the explosives which

32/ Bureau of Mines, Procedure for Testing Explosives (Including Sheathed Explosives) and Blasting Devices for Permissibility and Suitability: Sched. 1F, approved January 20, 1945, 11 pp., with amendments approved Mar. 14 and Aug. 7, 1945, and June 24, 1946.

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passed the tests was, at the request of the manufacturer, placed directly on the inactive list.

Substitutions in Permissible Explosives

A great variety of materials is used in compounding permissible explosives. Changing conditions and demands of the chemical industry have made many of these substances very difficult or virtually impossible to obtain. It has, therefore, been necessary for manufacturers to seek safe and suitable substitutes for some of the ingredients entering into the composition of these explosives. Seven permissible explosives containing proposed new ingredients have been tested for approval.

A permissible explosive approved originally in two different cartridge diameters was resubmitted in six additional diameters to establish the basic data for these sizes.

Field Samples of Permissible Explosives

A permissible explosive or blasting device, after approval for use in gassy and dusty coal mines, must be so manufactured that it will have all the chemical and physical characteristics of the basic sample. For a check on their quality, 18 samples of permissible explosives were collected from the manufacturers' magazines. All samples passed the gallery tests, although several did not meet the established tolerances of some of their physical characteristics. The items in which these explosives failed, and the number of failures respectively, were as follows: Physical examinations, 2; chemical analyses, 8; rate of detonation, 5.

Consumption of Permissible Explosives

During the fiscal year 1947, the consumption of permissible explosives in coal mines was approximately 110 million pounds. In addition to this, approximately 33 million pounds of black blasting powder and approximately 119 million pounds of high explosives other than permissibles were used, chiefly in strip-mining operations. Thus, over three and one-half times as much permissible explosives as black blasting powder was used. This ratio has been steadily increasing since 1940, the first year in which the consumption of permissible explosives exceeded that of black blasting powder.

Firedamp Safety of Powder-Powered Tools for Splicing Cables and Punching Rails

It is to be anticipated that demands for increased production of coal will result in the production of special tools for performing tasks in coal mines with greater ease and efficiency. Tools for splicing cables and punching rails have recently been placed on the market. They are powered by cartridges containing requisite amounts of propellent powder.

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Tests have been made to determine the firedamp safety of such tools.[33] They have been tested under a variety of conditions in a mixture of natural gas and air. The studies suggest that employment of these tools in a normal functional manner under the conditions outlined involves very little, if any, ignition of firedamp. A total of 83 gallery tests of the cable splicer, including 33 under drastic conditions emphasizing ignition hazards, and a total of 60 gallery tests of the rail punches were made without ignition of 8 percent mixtures of natural gas and air. Some hazard was found to attend some of the ammunition supplied for the rail punch. When fired directly into natural gas and air, the ignitions were obtained with the cartridge alone. This ammunition can be modified to minimize such hazards.

Determination of the Reactivity of Calcium Carbide and Acetylene-Water Vapor Mixtures

In studies made by the Bureau of Mines to determine the causes of explosions in medium-pressure acetylene ganerators, it was shown[34] that, when these are operated at elevated temperatures, the acetylene generator carries enough water vapor to heat certain types of carbides to a high temperature, when the acetylene-water vapor mixtures are passed through the carbide. It was found that reproducible results on the test characteristics of carbides could not be obtained in a small laboratory-type apparatus. Accordingly, a larger testing apparatus was designed which more closely simulated conditions prevailing in a commercial generator. It consists of a 20-pound portable acetylene generator, suitably altered to allow the desired tests to be made. Details of the apparatus and procedures are described elsewhere.[35]

The acetylene-water mixture is passed through the test carbide for 4 hours, or less if the test carbide heats to a maximum and then cools down in all three thermocouple locations before the 4-hour period has expired. Experience has shown that a highly reactive carbide will develop its maximum temperature within the time limit of 1-1/2 to 2-1/2 hours under the experimental conditions described. Low reactive carbides may require 4 hours to develop a maximum temperature.

Typical heating curves of different carbides, namely, low reactive, moderately reactive, and highly reactive, were obtained. In all cases the temperature of the generator water was 65° C., and thus the saturated acetylene had a water vapor content of 24.7 percent by volume. For low-reactive carbide, the maximum temperature reached in any part of the charge during

33/ Denues, A. R. T., Powder-Powered Tools; The Firedamp-Safety of Devices for Splicing Cables and Punching Rails: Bureau of Mines Rept. of Investigations 4105, 1947, 11 pp.

34/ Jones, G. W., Scott, G. S., Kennedy, R. E., and Huff, W. J., Explosions in Medium-Pressure Acetylene Generators: Bureau of Mines Rept. of Investigations 3755, 1944, 20 pp.

35/ Jones, G. W., Scott, G. S., Kennedy, R. E., and Spolan, I., Apparatus for Determining the Reactivity of Calcium Carbide and Acetylene-Water Vapor Mixtures: Bureau of Mines Rept. of Investigations 4067, 1947, 15 pp.

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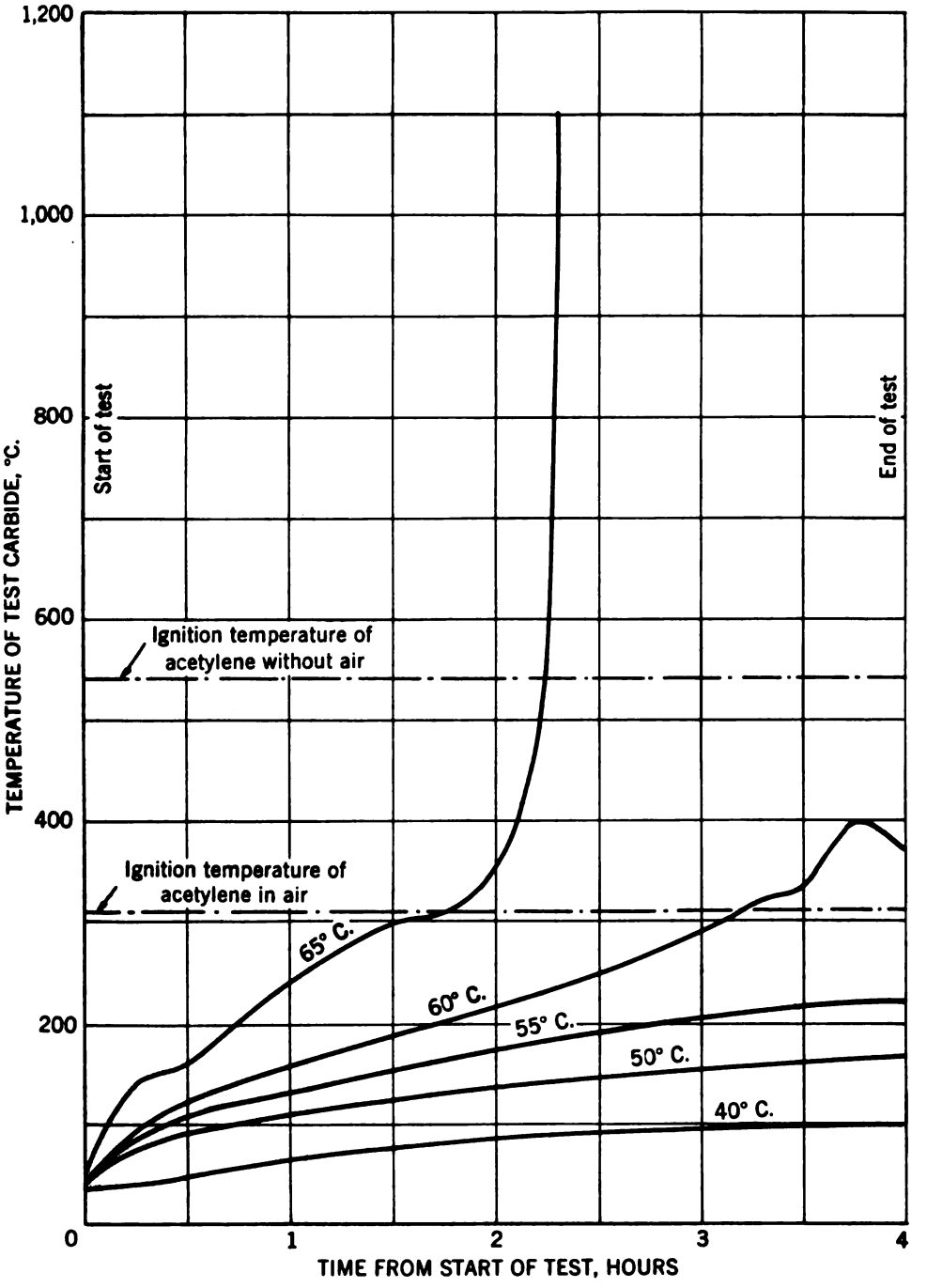

Figure 32. - Effect of temperature of water in generation on heating of carbide. Highly reactive carbide; size, 1/4 x 1/12 inch; generator pressure, 4 lb. per sq. in. gage.

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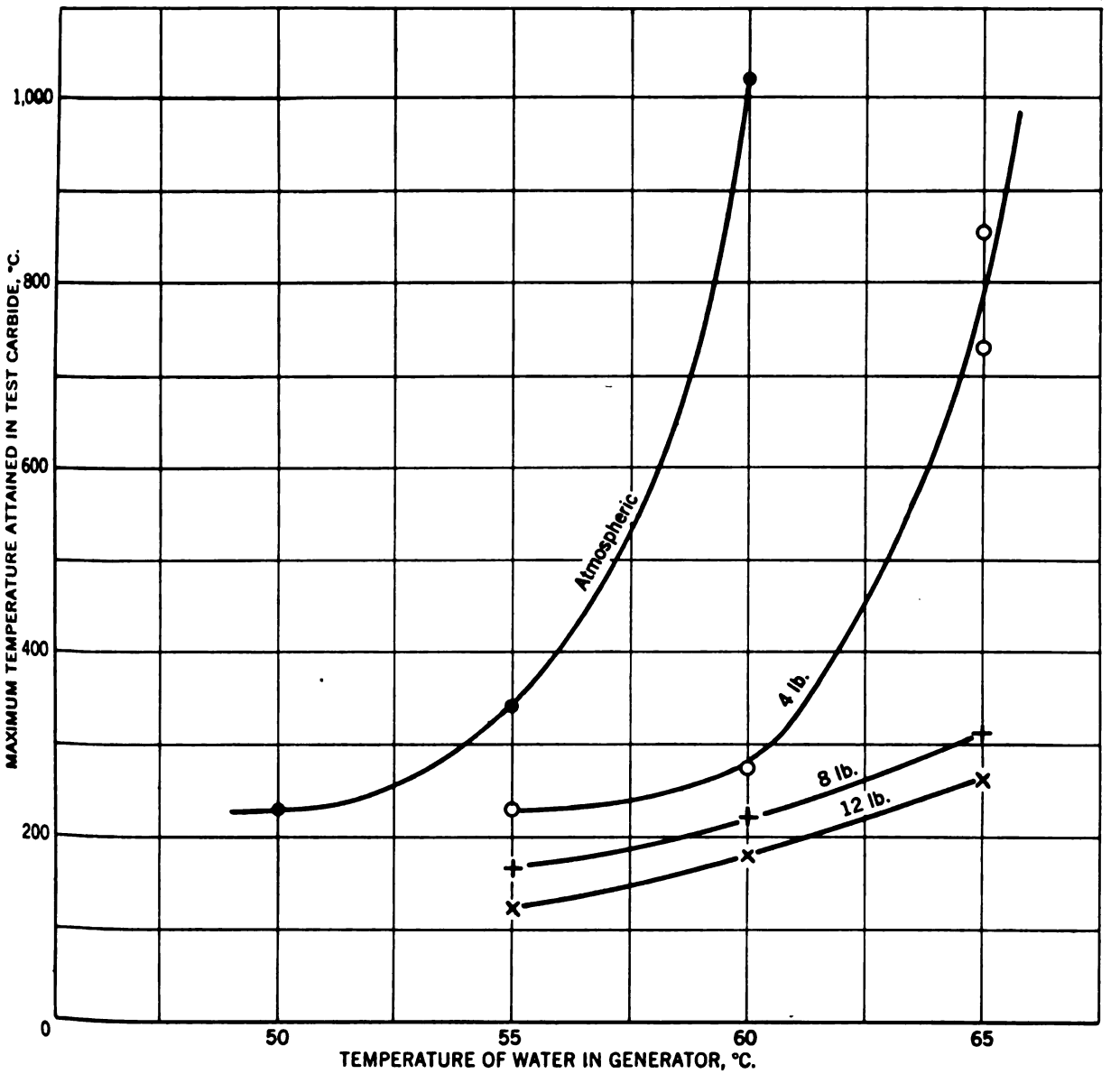

Figure 33. - Effect of generator water temperature and pressure on reactivity of carbide.

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the 4-hour period never exceeded 230° C., which is well below the ignition temperature of acetylene and air (310° C.). For moderately reactive carbide, the temperatures rise somewhat above the ignition temperature of acetylene in air 2 hours after the start of the test, particularly in the middle and top part of the charge. In the top part of the charge it reaches a value of 406° C. 2-1/2 hours after the start of the test and then drops abruptly to below the ignition temperature. The bottom part of the charge remains safely below the ignition temperature of acetylene in air throughout the 4-hour test. For highly reactive carbide, very rapid rise in temperature occurs after about 1 hour; after 2 hours it rises at a rate of 100° C. per minute and attains temperatures well over 800° C. in about 2 hours. The temperature of the top part of the charge rises earlier than the rest of the charge. This rapid rise in temperature is probably due to heat liberated in the decomposition of acetylene alone (ignition temperature, 540° C.).

Using 1/4- by 1/12-inch carbide, tests were made of the effect of generator-water temperature, generator pressure, and rate at which acetylene gas is passed through the carbide on the heating effects.

The generator-water temperature was varied from 40° to 65° C., the pressure being maintained at 4 pounds above atmospheric and acetylene rate at 32 cubic feet per hour. The carbide used was known to heat excessively in contact with water vapor. The maximum temperature attained in the carbide increased with generator water temperature in a manner shown in figure 32. It is clear from these results that a dangerous condition is approached at generator-water temperatures above 55° C. for this type of carbide.

Typical results showing the effect of pressure on the reactivity of a highly reactive carbide are given in figure 33. It is seen that, the lower the pressure in the generator, the higher the temperature attained, other conditions of the test being the same. For highly reactive carbides change of generator pressure had a very marked effect upon the temperature attained in the test carbide; for low-reactive carbides it had only a moderate effect. The fact that increasing the generator pressure reduces the maximum temperature attained by the carbide indicates that possibly the heating is governed by the water-vapor content because at a given temperature of the generator water, the percentage of water in the mixtures falls off with increasing acetylene pressure.

The results obtained with different flow rates of acetylene show that the temperature of the carbide increases with increasing flow rate, reaching a maximum at about 32 cubic feet per hour. Above this rate, the temperature falls again, except for the highest-reactivity carbides; and here the additional rise in temperature is small - up to 39 cubic feet per hour.

On comparing the results for various brands of carbide obtained to date, it becomes evident that particle size is not the only factor contributing to heating. It is probable that surface reactivity plays an important role.

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PREPARATION OF COAL

Recovery of Fine Coal from Slurry Water

An economical method for recovering fine-size coal, on a limited scale, from slurry water that was previously wasted has been developed and installed at one of the mines in the Warrior field of Alabama.36/ The method consists principally of recovering the fine coal by pneumatic froth flotation and using the washed-coal bin as a drainage and filtering medium for recovering the fine coal out of the froth. Under normal operations, the system will recover almost 2 tons per hour of coking coal out of slurry that was previously wasted.

Progress is being made in the development of a kerosine flotation process for cleaning and dewatering fine sizes of coal from about 10-mesh to 0. A four-cell semicommercial machine has been obtained for the laboratory and for experimental work in commercial plants. Results with this machine and a larger four-cell machine installed by one of the cooperating companies have established quite definitely the feasibility and practicability of the process for treating underflow sludges from dewatering screens. Efforts are now being directed toward making the process applicable to the treatment of raw-coal fines.

Preparation Characteristics of Illinois Coals

An intensive study of the preparation characteristics of the four principal beds in Illinois - namely, the No. 2, No. 5, No. 6, and No. 7 - was undertaken. Face and tipple samples from mines in each of the several producing districts were float-and-sink tested, and sieve analyses of the slack coals were prepared. Although all the field work has been completed during the fiscal year, the final analysis and interpretation of test data have not been accomplished.

New Heavy-Medium Process for Cleaning Fine Coal

The various heavy-medium processes in use for separating the impurities from raw coal as mined are not suitable for cleaning fine sizes of coal. The particular advantage of processes that employ a suspension of solid material in water or a heavy liquid to effect a separation between coal and impurity, based alone on differences in specific gravity, is that they are able to make a more complete and efficient separation than processes that depend also on other physical properties. Unfortunately, however, heavy-medium processes cannot ordinarily be used to clean the fine sizes of coal below 1/4 inch.

As a result of observations made by members of the wartime Solid Fuels Mission at the Netherlands State Mines in Europe this situation has changed.

36/ Gandrud, B. W., and Riley, H. L., Flotation Treatment of Washery Water at the Empire, Alabama, Mine of the DeBardeleben Coal Corporation: Am. Inst. Min. and Met. Eng., Tech. Pub. 2205, 1947, 8 pp.

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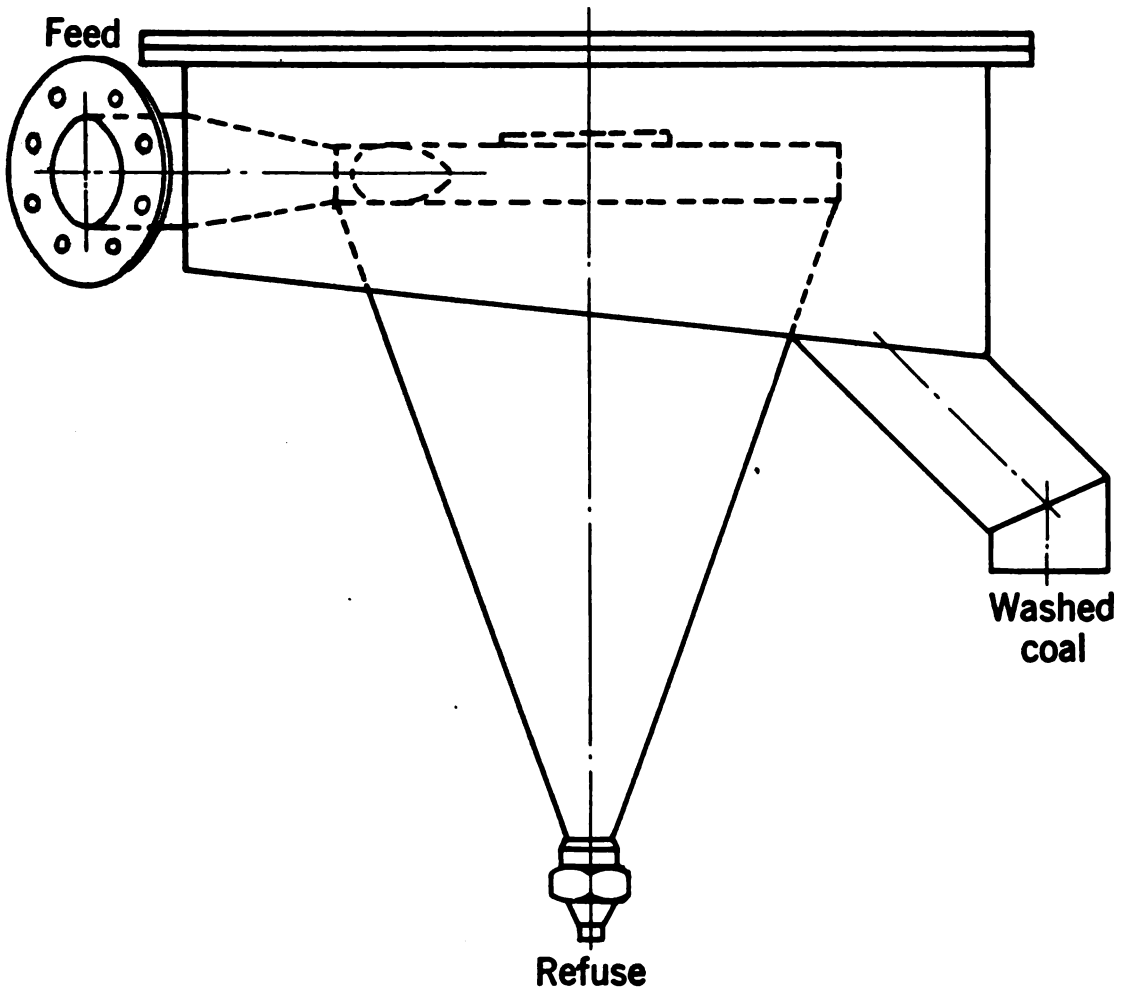

Figure 34. - Netherlands State Mines cyclone washer for fine coal.

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A cyclone-type coal washer for fine coal was developed at the State Mines, Heerlen, The Netherlands, during the German occupation, and the information concerning it has been brought back to the United States. Since then the Northwest Experiment Station of the Bureau at Seattle, Wash., has built one of these Netherlands cyclones of laboratory size and studied its performance as a cleaner of fine coal. The results of this preliminary investigation were presented at the joint fuel conference of the American Institute of Mining and Metallurgical Engineers and American Society of Mechanical Engineers held in Philadelphia, Pa., in October 1946, and have since been published.37/

Figure 34 shows a sketch of the cyclone washer. The 14-inch diameter size has a capacity of about 16 tons of feed coal an hour.

In appearance and operation the cyclone device looks much like the ordinary cyclone dust collector, except that it is operated wet. In shape it is an inverted cone, with an open, spigot-type discharge at the bottom and an overflow through an orifice at the center of the cone at the top. The coal feed, in a suspension of barite, loess, flotation refuse, or other high-gravity liquid, is introduced tangentially at the top of the cone along the side. The pulp density is about 6 of suspension to 1 of coal. In effect, this cyclone cleaner acts as a device for separating continuously the float-and-sink particles present in a bath having a density intermediate between coal and impurity. The coal feed must be suspended in a heavy medium.

Table 7 shows the results of a trial run made of coal from a mine in the Pittsburgh bed at Bruceton, Pa., crushed to pass 14-mesh. The results are summarized according to the individual size fractions present.

TABLE 7. - Summary of results obtained in cyclone test on 14- to 100-mesh Bruceton coal1/

Data	Screen size, mesh					
	14-35	35-48	48-65	65-100	-100	14-100
Screen analysis of feed .... percent	66.3	10.2	7.5	4.9	11.1	88.9
Sink 1.60 sp. gr. in feed .... do.	4.6	4.6	4.6	5.5		4.7
Yield of refuse ..... do.	4.9	5.0	6.5	9.0		5.3
Specific gravity of separation	1.60	1.67	1.68	1.88		
Sink 1.60 sp. gr. in washed coal ... .percent	.2	.9	1.2	2.4		.5
Float 1.60 sp. gr. in refuse . do.	9.6	25.7	47.2	62.1		20.8
Ash in feed ..... do.	7.4	6.8	6.8	7.4	6.5	7.3
Ash in washed coal ..... do.	4.8	4.6	4.8	5.6		4.9
Ash in refuse ..... do.	57.8	48.0	35.0	26.2		51.0
Efficiency ..... do.	99.7	98.8	96.8	93.6		99.0

1/ Barite suspension, 1.40 sp. gr.; feed-tube opening, 7/16 in.; washed-coal orifice, 7/16 in.; refuse opening, 1-3/32 in.; operating pressure, 20 psi.

37/ Goer, M. R., and Yancey, H. F., Preliminary American Tests of a Cyclone Coal Washer Developed in The Netherlands (with discussion): Am. Inst. Min. and Met. Eng., Tech. Pub. 2136, February 1947, 20 pp.

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In general, these results show a very efficient separation. The efficiency on all sizes combined, from 14- to 100-mesh, was 99.0 percent. The finer sizes of coal were separated at higher specific gravities than the coarser ones. The quantity of heavy impurity remaining in the washed coal increased with decreasing particle size. Similarly, the quantity of coal lost in the refuse product increased with decreasing particle size. Thus the efficiency of the separation between coal and impurity decreased with decrease particle size. Only the material coarser than about 48-mesh was handled with satisfactory efficiency. Below that size, the sharpness of the separation dropped rapidly.

The work showed that the cyclone has a high capacity and is able to make a sharp, efficient separation between coal and impurity at a particle size down to about 48-mesh. Thus the cyclone offers promise of extending substantially the size range that may be effectively treated by heavy-medium processes.

European Practice

Coal Preparation in The Netherlands

Current developments in the field of separation by heavy suspensions constitute the most interesting European development, because of present activity in this direction in America. It will be recalled that the practical application of this principle to the separation of minerals started with the introduction of the Chance sand-flotation process and large-scale experiments with the Conklin process in the Pennsylvania anthracite region during and immediately after the First World War.

In The Netherlands, three heavy-medium processes have been perfected and reduced to practice. Like our American processes, these differ primarily in the method of maintaining a controlled dispersion of the suspended solids.

The Barvoys process is the most widely used at present in Europe. The preferred medium for this process is a mixture of barite and clay of a size consist that will settle very slowly in water at operating gravities. It may be considered a semistable suspension. In this process, Conklin's problem of recovering the dispersed medium of the rinse water has been solved by exploiting the phenomenon that, with properly controlled size consist, a very fine medium will settle very slowly at the high concentrations used for separations and yet will settle rapidly in very dispersed suspensions, such as rinse water.

In contradistinction to this, the principle of the Tromp process uses a finely ground but yet definitely settlable magnetite suspension, which is just allowed to settle naturally in traversing the separating bath. Control involves only preknowledge of the rate of settling, so that the effective separation gravity may be predetermined. To avoid blocking the machine by accumulation of middlings in the bath, which naturally comes about with a difference in specific gravity between the top and the bottom areas of the medium, the material in the middle-depth portion of the bath is continually removed and reprocessed elsewhere in the plant.

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The Netherlands State Mines or loess process grew out of the difficulties experienced in obtaining such special materials as barite and magnetite. The scraper-discharge type of separator common to all the European heavy-medium processes is modified in The Netherlands process to shallow depth, with the top and bottom scraper lines close enough together to furnish mechanical agitation affecting virtually the entire content of the vessel. By this means rather unstable media of varying size consist are kept in suspension, and there is no place for large accumulation of middle products. Virtually all the material is drawn at once into one or the other of the two scraper lines. In practice, the gravity of separation is adjusted so that all the doubtful material goes with the refuse and subsequently is recovered in a separate middlings rewash operation.

Perhaps the most important fact about these processes for us is that a number of plants in successful operation in Europe and Great Britain demonstate the feasibility of using semistable suspensions.

Additional information on coal-preparation practice in Germany and The Netherlands is given in a report previously published.38/

Coal Preparation in Western Germany

A survey was made of coal-preparation practices in western Germany to discover any technological or organizational developments that might be of value to the American coal industry; to discover any special devices or techniques that may have been developed to prepare special grades of fuel to meet emergency war needs; and to ascertain the grades of coal and the methods of their preparation for use in the synthetic liquid-fuels industry.

The coal-preparation industry in Germany is organized very much as in America, in that the erection of new plants and extensive alterations are generally handled on contract by specialized equipment manufacturers who also control very largely the exploitation of new technological developments.

In the comparison of contemporary German and American coal-preparation practices there is (1) the greater predominance of conventional jig washers in the German field (although the Baum jig is also the most popular washer in America); (2) presizing of coal before jigging, as compared to almost universal practice of jigging unsized coal in America; (3) treatment of fine coal on jigs in Germany, contrasted to use of tables, launders, and classifiers in America; (4) extensive use of froth flotation in Germany; (5) widespread use of the Chance sand-flotation process in America, with no direct counterpart in Germany, although there is a more definite trend in that country to the use of substantially stable suspensions; and (6) loading of the product through bins in Germany, compared with the practice of direct continuous loading of prepared grades from preparation units to transport facilities in American practice.

38/ Yancey, H. F., and Fraser, Thomas, Coal Preparation in Germany and The Netherlands: Am. Inst. Min. and Met. Eng., Tech. Pub. 2112, 1946, 14 pp.

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Although Germany was the greatest coal producer in continental Europe before the war, 95 percent of its energy requirements being supplied by solid fuels, it lacked liquid fuels, as well as other materials. One essential material, derived from crude oil, which was virtually unobtainable, was petroleum coke for making electrodes for aluminum reduction and other metallurgical operations. This lack of an adequate source of pure carbon made necessary the development of methods of obtaining it from coal. Three methods resulted and are as follows: (1) Solution of coal in organic solvents and removal of inorganic impurities by filtration; (2) a two-stage cleaning process, comprising of a heavy-medium separation in a suspension of magnetite, followed by grinding of the clean coal and its recleaning by froth flotation; and (3) a combination of cleaning by froth flotation and digestion of the product in a hot, dilute mixture of hydrochloric and hydrofluoric acids to give a final product containing only 0.5 percent ash, which was then coked in standard byproduct ovens.

The three commercial plants incorporating these means of ultra-cleaning were all heavily bombed during the war and were not in operating condition at the time of the survey. However, such data as could be obtained regarding the technological functioning and performance of these plants are published in some detail.39/

Drying Lignite

Experimentation on steam drying of lignite in the 25-ton-a-day pilot plant at Grand Forks, N. Dak., was continued. After several hundred tons of lignite and subbituminous coal were dried, it was proved that the dried fuel was susceptible to spontaneous combustion and that it was dangerous to ship the product in railroad cars without further treatment. Consequently, a study was made of some of the factors effecting the spontaneous heating of dried lignite. Two experimental bins were constructed, one of which simulated a railroad car. This was an open-bottom steel bin 5 feet square and 3 feet high. The bottom was not welded, and on one side a 2-inch opening extended the entire width, allowing air to enter as it does in a gondola railroad car. Ten thermocouples were installed in this bin, 5 on a plane 1 foot above the bottom and 5 on a plane 2 feet above the bottom, and were connected to a recording pyrometer. The other bin was the same size, but the sides and bottom were welded gastight, and thermocouples were placed 1 and 2 feet from the bottom. Natural lignite, dried with 400-pound-pressure steam, was then fed to the bins at various temperatures, and the tendency of the coal to ignite spontaneously was observed. Tables 8 and 9 summarize the results of these tests.

39/ Fraser, Thomas, and Driessen, M. G., Coal-Preparation Practice in Western Germany: Bureau of Mines Inf. Circ. 7389, 1946, 70 pp.

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TABLE 8. - Spontaneous combustion of steam-dried lignite in an open-bottom bin[1]

Test No.	Condition	Temperature, °F. (Average of 10 thermocouples)[2] Time, hours 0	10	20	30	40	50	60	70	80	90	100	Maximum final temperature, °F.	Minimum final temperature, °F.
3	Cooled for 18 hours[3]	39	43	48	53	59	65	70	75	73	72	4/70	77	46
4	Partly cooled[5]	61	91	118	126	126	121	111	109	104	100	4/96	99	59
5	Loaded from chute[6]	105	135	155	145	140	250	7/365					575	140
6	do.[6]	101	130	125	100	90	95	105	165	7/250			600	75
7	Filled manually[8]	105	160	150	135	125	115	105	100	95	90	4/85	75	65
8	Filled and packed manually[9]	97	165	190	180	185	200	7/295					560	245
9	Oil treatment filled manually[10]	120	175	165	160	165	7/200						535	90
10	Mixed oil treatment filled manually[11]	108	165	150	140	135	130	125	120	115	115	4/110	125	60
11	do.[11]	105	180	160	155	150	165	210	7/300				645	105

1/ Steel bin 5 x 5 x 3 ft. not welded at bottom. One side of bottom provided with 2-inch opening extending full length of bin, simulating railroad car.
2/ 5 thermocouples on plane 1 foot from bottom, and 5 thermocouples on plane 2 feet from bottom.
3/ Spread on floor, 6 inches deep, cooled 18 hours and shoveled into bin.
4/ Did not ignite.
5/ Each batch spread on the floor, 6 inches deep; after the 7th batch all coal was shoveled into the bin.
6/ Each batch run directly into bin and leveled.
7/ Discontinued; ignited.
8/ Each batch was run on floor and immediately shoveled into bin. Prevents continuous segregation.
9/ Each batch was run on floor and immediately shoveled into bin and manually tamped.
10/ Each batch was run on floor, sprayed with 11 lb. No. 2 fuel oil, and immediately shoveled into bin.
11/ Each batch was run on floor, sprayed with 11 lb. mixed oil (50 percent No. 2 fuel oil and 50 percent lignite-tar oil), and immediately shoveled into bin.

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TABLE 9. - Spontaneous combustion of steam-dried lignite in a closed-bottom bin[1]

Test No.	Condition	Temperature, °F. (lower center, 1 ft. from bottom)						Temperature, °F. (upper center, 2 ft. from bottom)					
		Time, hours						Time, hours					
		0	20	40	60	80	Final	0	20	40	60	80	Final
4	Partly cooled[2]	50	72	106	128	[3]/126	98	50	81	128	155	[3]/150	113
6	Loaded from chute[4]	70	90	185	[5]/220		220	70	105	195	[5]/220		220
7	Filled manually[6]	95	135	150	[5]/140		125	95	150	190	[5]/205		220
8	Filled and packed manually[7]	110	140	200	[5]/205		405	110	140	200	[5]/400		540
9	Mixed-oil treatment[8]	105	110	195	[5]/185		385	105	130	195	[5]/240		585
10	do.[8]	105	110	195	[5]/180		185	105	145	200	[5]/250		495

1/ Steel bin 5 x 5 x 3 ft. open at top and welded on all seams.
2/ Each batch spread on floor 6 inches deep; after the 7th batch all coal was shoveled into the bin.
3/ Did not ignite.
4/ Each batch run directly into bin and leveled.
5/ Discontinued; ignited.
6/ Each batch run on floor and immediately shoveled into bin. Prevents continuous segregation.
7/ Each batch run on floor and immediately shoveled into bin and manually tamped.
8/ Same as test 7; in addition, each ton treated with 11 lb. mixed oil (50 percent No. 2 fuel oil and 50 percent lignite tar oil).

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In all of the tests with the open-bottom bin in which the coal finally ignited, the maximum temperature increased rapidly during an initial period of from 5 to 15 hours, usually to the range of 185° to 200° F., then decreased or remained approximately constant for an additional 25 to 35 hours, after which it rose rapidly to above 500° F. All of the 10 thermocouples followed this general pattern. In the tests in which dried coal did not ignite, the temperature of all thermocouples rose rapidly for the first 5 to 8 hours, then decreased very gradually.

In all of the tests with the closed-bottom bin, the temperatures increased more or less uniformally for the first 30 to 40 hours to the range of 180° to 200° F., then they leveled off for a brief period or rose rapidly to ignition and burning temperatures. In all cases, the burning started in the upper section of the bin.

Further work remains to be done; however, it is indicated that adequate initial cooling of the steam-dried lignite will prevent spontaneous ignition.

STORAGE OF COAL

In 4 years of operation, during which 30,000 tons of slack coal were utilized, it was demonstrated that storage in large open pits is feasible, with a loss of heating value as low as 1.5 percent in 1 year.40/

As given in the above publication, the following general points should be considered:

1. The walls of the storage pit should be tight, preferably made of concrete, or excavated in dense earth or clay.

2. It is important to avoid segregation of sizes in the coal as it is being unloaded or placed in the pit. The accumulation of zones of fine and coarse coal provides air entry courses which will supply oxygen to the interior of the stored coal. The entire mass of stored coal should be as homogenous as possible.

3. In placing the coal in the pit, care should be taken to store it in shallow benches 2 to 3 feet in depth. Observations have shown the latter to be both practical and economical. Each bench should be leveled and compacted to as high a packing density as is practical. A bulk density of 56 pounds per cubic foot can be attained readily with slack coal.

4. It has been demonstrated that uniform compaction of the pit should be practiced, taking care of the marginal edges as well as the inner areas. Remnants of old coal remaining from the previous storage should be spread out in thin, uniform layers over the bottom of the pit before benches of new coal are placed in storage. Observations have revealed that snow, weeds, and other debris, which have accumulated on the bottom of the pit or on

40/ Goodman, John B., Parry, V. F., and Landers, W. S., Storage of Subbituminous Slack Coal in Open Pits: Bureau of Mines Rept. of Investigations 3915, 1946, 37 pp.

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benches of coal, should be removed before any new coal is placed, since they preclude dense packing and eventually become loosened void spaces which enable air to circulate.

5. The finished stored coal should be level in appearance and, if possible, flush with the top edge of the pit walls to avoid entrainment of wind currents. Valleys and peaks appearing on the surface of the storage pile should be leveled, because irregularities will catch wind currents and will induce air circulation to cause self-heating.

6. In a pit with tight side walls, air can enter only through the top surface. To impede movement of air into the coal mass, the surface should be compacted by rolling or scraping to break down the top few inches of slacked coal into fines.

UTILIZATION OF COAL FOR COMBUSTION

Fuel-Engineering Service

Fuel-engineering service to Government establishments in the inspection, selection, and use of fuel and fuel-burning equipment was continued. At the request of the Veterans Administration, total operating-cost comparisons were made when oil, gas, and coal are used, and the type of fuel-burning equipment was recommended for 38 new projects; the complete design of a new boiler plant at Fresno, Calif., was studied, and recommendations were made for improvement. At the request of the Federal Works Agency, a study was made of boiler plant changes for improving operation at the Lexington, Ky., Marine Hospital. At the request of the Federal Housing Agency, fundamental designs for two types of proposed standard boiler plants were planned and recommended. At the request of the Federal Public Housing Administration, recommendations were made on changes in operation and in boiler furnace and baffles at its Springfield, Ill., project, where external corrosion of boiler tubes was causing excessively high maintenance costs. At the request of the Public Buildings Administration, a study was made of revamping the Washington, D. C., Government fuel yard coal-storage system. Inspections were made of four Federal Housing projects in the vicinities of Philadelphia, Pa., and Newark, N. J. Recommendations were made on the control of corrosion difficulties and on the handling and burning of the fuel. Service on 65 special problems was given 24 different Government agencies. Although the war project, "The National Fuel Efficiency Program," was terminated at the close of the war, requests have been continually received for hundreds of copies of the literature and posters used during that program for improving fuel utilization. Consulting service as to the purchase of fuel appropriate for the equipment was given the Justice Department and the Office of Indian Affairs for all of their plants, and also for a number of plants of various other agencies. A representative of the Bureau of Mines served as chairman of the interdepartmental Federal Fuel Purchasing Committee created to establish Government policies on fuel purchases; he also served as chairman of the Technical Committee on Solid Fuels, created to prepare standard Federal specifications for solid fuels. Work with the American Society for Testing

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Materials included the chairmanship of a section "Combustion of Coal and Coke," created to prepare a write-up covering the significance of tests of coal and coke in relation to combustion in different types of fuel-burning equipment.

Cooperative work with the Air Preheater Corp. on research on the prevention of deposits and corrosion of boiler air preheaters was continued. Laboratory tests are being run, and field-test and operating data from many installations throughout the United States are being accumulated. Different materials and changes of operation are being studied. Substantial progress is being made in improving operation and lowering maintenance cost.

Boiler Feed-Water Conditioning

Analyses and resulting recommendations were made on 9,129 samples of boiler water during the fiscal year, as follows: 6,388 from the War Department; 1,095 from the Veterans Administration; 476 from District of Columbia plants; 294 from the Post Office Department; 284 from the National Housing Agency; 260 from the Office of Indian Affairs; 184 from the Department of Justice; 55 from the Public Health Service; 29 from the Department of Agriculture; 27 from the Navy Department; 19 from the Department of Commerce; 9 from the Federal Works Agency; 6 from the Atomic Energy Commission; and 3 from the War Assets Administration. Special analyses and recommendations were made on 7 samples of several types of water for various agencies. Reports and recommendations covering 22 analyses of various scales, sludges, and deposits and 18 covering analyses of boiler compounds were made. An average annual saving of about $240.00 was effected by the use of standard chemicals instead of proprietary compounds at several National Housing Agency plants. Two hundred and seventy eight special Bureau of Mines field-water test kits, 9,000 bottles of chemical reagents, and 10,517 test-kit replacement items were distributed to various Government activities. Visits were made to 189 heating plants operated by a number of Federal agencies in the local area of the District of Columbia to advise on boiler-water treatment, give instructions in boiler-water testing, and inspect boilers for scale and sludge.

At the request of the National Advisory Committee for Aeronautics, a visit was made to Langley Field, Va., to give instructions on building chemical feeding equipment. As a result of the visit, a saving of $3,000 was effected in the cost of feeding equipment; in addition, silica jell used for protection of idle boilers was replaced by quicklime, a much cheaper material. At the request of the Navy Department, instructions on boiler-water conditioning were given to personnel at the U. S. Naval Torpedo Station in Alexandria, Va., and the U. S. Naval Torpedo Testing Range in Piney Point, Md. At the request of the District Government, instructions were given to District Government Headquarters' engineers on boiler-water testing and on boiler-water treatment and control. A report was made to the District Government of an acceptance test of a zeolite softener at Gallinger Hospital. Consulting service was given the Public Buildings Administration on control of corrosion in return lines; the Veterans Administration in preparing a manual on boiler-water treatment and on boiler-water testing; the Capitol Power Plant on boiler-tube

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failure; the Bureau of Yards and Docks, Navy Department, on writing a manual on boiler-water treatment; and the Second Army Headquarters on boiler-water treatment. At the request of the Federal Public Housing Authority, plans were completed for extending the boiler-water-conditioning service to Federal housing plants throughout the United States.

Instructions on Testing of Boiler Water

An instruction booklet[41] on testing for tannin by boiler-water color was revised and printed, with illustrations. A pH test for water from low-pressure boilers by the use of indicator paper was developed, and directions for the test were completed.[42] An instruction booklet[43] for testing boiler water for phosphate and causticity was rewritten and revised, with illustrations.

Innovations in Boiler-Water-Control Testing

A comparative study was made of the determination of total dissolved solids in boiler water by hydrometer, electrical conductivity, and evaporation to dryness. The electrical conductivity method was not found to be superior to the hydrometer in accuracy for routine samples coming from widely dispersed sources. The stability of the standard phosphate solution was tested and a procedure determined for increasing the stability in the type of bottle used. Comparison was made of changes in the silica content of boiler-water samples in rubber and glass bottles to determine methods of bottling boiler-water samples for determination of silica. Study was continued, throughout the year, of the determination of chloride in boiler waters containing tannin, using the Mohr titration, that is, titration with silver nitrate, using a chromate indicator. The presence of the tannin obscures the end point. Also, high concentrations of phosphate, which may be present in boiler-water samples, interfere with the accuracy of the test. A procedure is being worked out for removing the tannin by decolorizing carbon for this test and for making necessary adjustments to eliminate interference of phosphate. Work was published[44]

41/ Bureau of Mines, Boiler-Water Service Section, Instructions for Bureau of Mines Boiler Water Test Kit for Tannin by Boiler Water Color: Form BWS9.

42/ Bureau of Mines, Boiler-Water Service Section, Bureau of Mines pH Test for Boiler Water from Low Pressure Boilers by Use of Indicator Paper: Form BWS14.

43/ Bureau of Mines, Boiler-Water Service Section, Instructions for Bureau of Mines Boiler Water Test Kit for Phosphate and Causticity: Form BWS1.

44/ Goldman, L., and Love, R. N., Colorimetric Phosphate Tests for Boiler Waters Containing Tannin: Bureau of Mines Rept. of Investigations 3983, 1946, 11 pp.
Goldman, L., and Love, R. N., Colorimetric Phosphate Tests for Boiler Waters Containing Tannin: Power Plant Eng., vol. 50, No. 1, 1946, pp. 76-79.
Goldman, L., and Love, R. N., Testing Boiler Water: Heating and Ventilating, vol. 44, No. 1, January 1947, p. 110.
Goldman, L., and Love, R. N., Tests for Boiler Water Containing Tannin: Combustion, vol. 18, No. 7, January 1947, p. 44.
Goldman, L., and Love, R. N., Tests for Boiler Waters Containing Tannin: Chem. and Eng. News, vol. 25, No. 5, February 3, 1947, p. 325.

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on the determination of phosphate in boiler water containing tannin. Three procedures were studied: (1) Coagulation of sludge by heating with potassium nitrate and use of color blanks to cancel out the color of the tannin; (2) bleaching of the tannin with potassium persulfate; and (3) removal of the tannin with decolorizing carbon, a new method developed in our laboratories. A gravimetric A.S.T.M. method was adapted for use as a reference method for the three colorimetric methods. It was shown that values obtained by any of the three colorimetric methods agree. This indicates that calcium phosphate sludge can be filtered out completely, even though tannin is present in the sample, as in the potassium nitrate coagulation method. The new method (3) was shown to be the most rapid and convenient of the methods studied.

Boiler-Water Research

Research was continued on the use of amines in boiler water, which volatilize with the steam and destroy the carbon dioxide acidity of the condensate formed. When such condensate is returned to the boiler as feed water, its amine content is again volatilized with the steam. Loss in wasted condensate in blow-down and at vents must be replaced periodically. A compounded dye was developed to add to a sample of condensate so that the boiler operator would know when the proper range of amine content was being carried. A method of calculation was devised so that a close approximation of the quantity of amine required for treatment could be determined when the percentage of make-up and blow-down was known for a given water. Such calculations furnish quantity and cost data from which it can be estimated whether or not the cost of amine treatment would be economical. A field study of chemical treatment[45/] showed that three amines, cyclohexylamine, benzylamine, and morpholine, of widely different alkalinity and volatility prevented corrosion equally well when present in sufficient quantity to neutralize carbonic acid in the condensate. Cyclohexylamine is very volatile in boiling water and quite alkaline, benzylamine is moderately volatile and moderately alkaline, and morpholine least volatile and only mildly alkaline. High volatility tends to result in losses during deaeration; low volatility tends to result in losses in the blow-down. High alkalinity tends to prevent carbon dioxide removal, thereby increasing the percentage recirculation of this substance and its concentration in the system. The higher the concentration of amine required in the system, the greater the amine make-up required since, in general, losses are due principally to wasted condensate. At the particular plant tested, which was operated at a steam pressure of 65 pounds per square inch, cyclohexylamine was lost in appreciable quantity at the feed-water heater vent, as well as in the wasted condensate; blow-down losses were important only with morpholine; benzylamine averaged superior to the others in these respects, owing to its intermediate volatility. The morpholine content decreased during the distribution of the steam at low pressures (5 psi). With these data as background, suitable chemical treatment for preventing corrosion in condensate piping has been recommended for a large number of Government-operated heating plants.

45/ Bork, A. A., and Nigon, J., Amine Volatility and Alkalinity in Relation to Corrosion Control in Steam-Heating Systems: Bureau of Mines Tech. Paper; in press.

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Under the chairmanship of a representative of the Bureau of Mines, a subcommittee of the American Society for Testing Materials revised seven standard methods of analysis relating to boiler water.

Smoke Abatement

Civic interest in smoke abatement continued to increase throughout the year. Many cities made requests of the Bureau of Mines for publications and consulting service on smoke abatement. These cities included Omaha, Neb.; Grand Junction, Colo.; Clarksburg, W. Va.; Cleveland, Ohio; Fort Washington, Wis.; Boise, Idaho; Winnepeg, Canada; Oak Ridge, Tenn.; Tacoma, Wash.; Hagerstown, Md.; Pontiac, Mich.; Westmount, Quebec, Canada; and York, Pa. The chief smoke-regulation engineer of Atlanta, Ga., requested several hundred copies of a number of publications covering fuel-burning equipment operation for the purpose of distributing them to various boiler plants in Atlanta to aid in the abatement of smoke. At the request of the smoke-regulation engineer of the District of Columbia, a representative of the Bureau served as a member of a committee to investigate fly-ash complaints of property owners in one area of the city. At the request of the Office of the Chief of Ordnance, War Department, a 2-day survey was made of air-pollution difficulties at Morgantown, W. Va., the local Chamber of Commerce having complained that the Morgantown Ordnance Works was the source of excessive pollution of the city air. A visual survey of the principal area of the city and an inspection of the plant in question were made. Certain changes in plant operation were recommended, and means were suggested for determining the contaminants of the air over a period of time. A report, entitled "Tentative Example Sections for a Smoke-Regulation Ordinance," was completed by the American Society of Mechanical Engineers Smoke Law Committee, of which a Bureau of Mines representative is chairman. Hundreds of copies of this report have been requested by many different interests.

Determination of Solid-Fuel Reactivity by Ignitibility

The absolute reactivity of a solid fuel is an extremely important property for evaluating the behavior of coal between the extremes of the slow rate of combustion occurring in a storage pile and the high specific rate of combustion of pulverized coal in a gas-turbine combustor. To date, however, no method for determining reactivity will give an unequivocal value, for any given fuel, that can be correlated with the behavior of that coal in storage or burned on a grate or in pulverized form.

To investigate the many factors that affect the determination of reactivity by the ignition-point method, for the purpose of developing a reliable and simple procedure, numerous tests were run with coal from the Pittsburgh seam in the conventional ignitibility apparatus. It has been found that the reproducibility of results in the same apparatus and the agreement of results with different apparatuses of the same design were improved considerably by suitable placement of the thermocouples. Also, it has been found that the order of the reaction with respect to oxygen concentration is less than 1, and that the reactivity is directly proportional to the specific surface of the sample.

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In measuring the reactivity by the ignition-point method, it is essential that the thermocouples be resistant to corrosion, generate relatively large voltages, and be made of uniform thermo-elements to secure reproducibility. Chromel-alumel elements, which possess the first two of these properties, were tested thoroughly for uniformity. It was found that a sulfur-vapor bath affords an excellent means for calibrating couples. Additional annealing improves the quality of 28-gage "standard-oxidized" wire, and the "bright-annealed" wire is quite uniform at low temperatures, but unstable.

External Corrosion of Furnace-Wall Tubes

This research has been extended to obtain quantitative determinations of the effect of temperature and concentration of SO_3 on iron oxides, alkali-metal sulfates, mixtures of iron oxide and alkali-metal sulfates, metallic iron and aluminum, and these metals in contact with synthetic enamel-type deposits.

In addition, experiments have been made to determine the quantity and the kind of gases evolved from coal ash during sintering and slagging processes. This phase of the investigation was conducted to confirm the mechanism of attack of furnace tubes that are covered with deposits which are predominantly alkali-metal sulfates - the so-called enamel-type, which is a term descriptive of the appearance of these deposits on the tubes shortly after the furnace is shut down.

Experiments have been conducted to determine the mechanism of attack of furnace tubes when the deposit consists mainly of iron sulfide, magnetic iron oxide, and carbon. Attack of tubes under these conditions has been dubbed "sulfide-type" to distinguish it from that which occurs when sulfate deposits predominate. On the assumption that pyrites from the coal, which adheres to certain tubes in certain furnaces, initiates the sulfide type of attack, some success in clarifying the mechanism has been obtained from experiments, under controlled laboratory conditions, with solid-solid reactions between iron sulfide and metallic iron, and iron disulfide and metallic iron.

Smokeless Packaged Fuel for Domestic Use

As the result of renewed interest and activity in smoke control in large cities, and the interest of domestic consumers of solid fuels in improved methods of hand-firing, an investigation was made of the burning characteristics of certain special packaged fuels containing low-volatile bituminous coal and asphaltic binder; high-volatile bituminous coal and binder; a mixture of the latter with a special "activator" composed of a liquid hydrocarbon and coal; and a mixture of high-volatile bituminous coal, low-temperature coke, and binder.

The experiments were conducted in a typical domestic hot-water boiler equipped for determining continuously the burning rates and smoke emission,

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and for fuel-bed photography, under controlled operating conditions. The characteristic receiving particular attention was the smoke produced by each blend, and for this purpose a specially designed photoelectric-type smoke meter was used to determine the factors important from the standpoint of smoke control - instantaneous smoke density and total relative smoke.

The total relative quantity of smoke produced during burning of the packaged fuels increased with the volatile content of the fuel.

The addition of low-temperature coke to high-volatile bituminous coal reduced the total relative quantity of smoke but resulted in lower burning rates, more ash-pit losses, and firing difficulties.

All the fuel mixtures studied produced coke strong enough to prevent crumbling in the fuel bed and resulted in burning rates high enough to meet domestic requirements.

High percentages of high-volatile bituminous coal in the packages required the use of secondary air to prevent excessive smoking caused by the formation of a plastic layer on the top of the fuel bed.

Mineral Wool from Rhode Island Anthracite

Eight tests were made with a slagging-type gas producer to secure slag from Cranston coal that would be suitable for the manufacture of mineral wool. To date, the operating experience for a wide variety of test conditions, such as various arrangements of the tuyeres and tap hole and different charges of limestone and coal, show that the initial period of the formation of slag and tapping is followed by displacement upward of the reaction zone in the fuel bed. This causes cooling of the shaft and the tuyere zone, so that tapping ceases within a relatively short time. It is believed that, although adequate tapping might be obtained merely by making minor changes in the arrangement of the tuyeres, tap hole, and floor of the producer, the flexibility of operation would be exceedingly narrow unless the blast air is preheated to at least 1,000° F. Accordingly, with the information from the tests that have been made, optimum arrangement of the component parts of the producer has been adopted; and an oil-fired preheater, designed to heat 2,000 pounds of air per hour to 1,200° F., is now under construction.

Developments in Switzerland

The economic situation with regard to fuels in Switzerland during recent years has resulted in theoretical studies and engineering developments that otherwise might not have been undertaken.[46] The high cost of coal led to turbine developments by three companies. The companies have an optimistic attitude regarding the solution of the abrasion problem in coal-fired gas turbines. Some combustion turbines, of Swiss design, were built in Germany.

46/ Rose, Harold J., Recent Engineering Developments in Switzerland on Gas Turbines and Steam Generators: Bureau of Mines Inf. Circ. 7403, 1947, 38 pp.

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An oil-fired turbine locomotive was tested and accepted in Switzerland, but its use was discontinued owing to the shortage of fuel oil. The heat pump has been used in Switzerland for space heating, air conditioning, industrial drying, and evaporation.

CARBONIZATION AND GASIFICATION

Laboratory Tests of Coking Coals

Small-scale laboratory carbonization tests made on various coals to evaluate their metallurgical coke-making properties as part of the survey of the gas- and coke-making properties of American coals include the United States Steel Corp. high-temperature distillation test, the Fischer low-temperature carbonization assay, and the Bureau of Mines agglutinating-value test. Technical papers[47] giving carbonization assays on 3 eastern and 28 western coals and agglutinating indices for 3 eastern and 89 western coals have been published. Agglutinating values of the western coals examined showed a better relationship to the coking quality of the coals than have those from Appalachian coals, which have shown poor correlation of agglutinating-value tests with shatter and tumbler tests of cokes made from these eastern coals. Most of the western coals examined are deficient in agglutinating materials, whereas many of the Appalachian coals have more of such materials than is required to form good coke. It is believed that agglutinating-value tests distinguish better between coals with a deficiency of such material than between those with an excess.

Agglutinating-value tests were made of 104 drill-core, outcrop, and mine samples of coal submitted in connection with investigations to determine minable reserves of coking coals for steel plants. The coals were from the Coosa field in St. Clair County, Ala., the Coal Creek district in Gunnison County, Colo., and the Georges Creek and Castleman Basin fields in western Maryland. Agglutinating values of drill-core and mine samples from the Coosa field in Alabama ranged from 5.2 to 12.0 kilograms and those of outcrop samples from 0.0 to 6.7 kilograms. The results indicate that many of the coals are strongly coking. Low values for most of the outcrop samples are probably due to oxidation of the coal by weathering. Agglutinating values of Castleman Basin and Georges Creek coals of Maryland ranged from 5.8 to 11.9 kilograms, which are satisfactory for coking coals. The Coal Creek coals of Colorado had agglutinating values ranging from 1.9 to 6.2

47/ Reynolds, D. A., Davis, J. D., Brewer, R. E., Ode, W. H., Wolfson, D. E., and Birge, G. W., Carbonizing Properties of Western Coals: Bureau of Mines Tech. Paper 692, 1946, 79 pp.
Reynolds, D. A., Davis, J. D., Ode, W. H., Wolfson, D. E., and Birge, G. W., Carbonizing Properties of Velva Lignite from Ward County, North Dakota, and Monarch Coal from Sheridan County, Wyoming: Bureau of Mines Tech. Paper 695, 1946, 41 pp.
Reynolds, D. A., Davis, J. D., Brewer, R. E., Ode, W. H., and Wolfson, D. E., Carbonizing Properties of Elkhorn No. 3-bed Coal from Wheelwright Mine, Floyd County, Ky.: Bureau of Mines Tech. Paper 697, 1947, 41 pp.

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kilograms. These results have been incorporated in a progress report[48] in which it is shown that coals occur in the Coal Creek district that are equal to the Sunnyside coal of Utah for making metallurgical coke.

Agglutinating-value tests on drill-core and mine sample of coal taken in connection with an investigation[49] of coal deposits in the Lookout Mountain field, DeKalb and Cherokee Counties, Ala., showed that many of the coals examined were strongly coking.

In connection with a survey of the effects of oxidation of coal during storage on the carbonizing properties of coking coals, agglutinating values of 15 coal samples were determined. The coals were from the Black Creek bed, Marion County, Ala.; Raton bed, Colfax County, N. Mex.; Pittsburgh bed, Allegheny County, Pa.; Eagle bed, Kanawha County, W. Va.; and Naricual Basin, Venezuela. From data that have been obtained on changes in agglutinating values of coals during storage, it was concluded that the agglutinating value is one of the best criteria of the extent of oxidation of stored coking coals.

Agglutinating-value tests were made on 29 samples of coal from Kentucky, Maryland, New Mexico, Pennsylvania, and West Virginia to determine whether these coals have sufficient coking properties to be suitable for export to blend with certain European coals for making metallurgical coke. The results showed that the coals ranged from weakly coking to strongly coking.

Agglutinating-value tests were made of six samples of coal from the Gorgas experimental mine, Pratt bed, Walker County, Ala., in connection with the preparation of this mine for experimental underground gasification tests. The values ranged from 6.9 to 8.5 kilograms, indicative of a quite strongly coking coal.

Eleven samples of Upper Cretaceous coal from northern Alaska were tested by the Fischer low-temperature carbonization assay to determine their value as a potential source of liquid fuel. The samples were collected in the summer of 1946 by the Federal Geological Survey in connection with Navy Department petroleum investigations in Naval Petroleum Reserve 4. The range of yields of carbonization products on an air-dried coal basis was as follows: Carbonized residue, 62 to 86 percent; tar, 9.6 to 37.6 gallons per ton; light oil, 0.85 to 2.7 gallons per ton; and gas, 1,200 to 3,360 cubic feet per ton. One of the coals formed a nonswollen coke that was very weak and friable. The carbonized residues from two samples were very slightly fused, with a few small cells. All of the other coals were noncoking in the Fischer assay, as carbonized residues were loose powders.

48/ See footnote 14, p. 22.
49/ See footnote 15, p. 26.

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Survey of Carbonizing Properties of American Coals

Studies on the carbonizing properties, plasticity, expansion, and oxidation of coal were continued. Only one coal was received for the complete series of tests by the BM-AGA method of determining gas-, coke-, and byproduct-making properties; it represented the Lower Banner bed and mine 56, Dante, Russell County, Va. Other investigations were made on nine high-volatile A coals that were tested mainly for yields of carbonization products in cooperation with the Republic Steel Corp., three samples of asphaltita for the Argentine Government, and one sample of coal for the Government of Venezuela. Further work was done on the small vertical expansion oven, and a paper for publication showing that it is not reliable for determining expanding properties of coal was prepared. Eleven coking coals previously tested for sensitivity to oxidation in the fresh state were carbonized after exposure in thin layers at room temperature (average, about 70° F.) for a maximum of 3-1/2 years.

Table 10 gives the analyses of all coals carbonized at 900° C. during the year, table 11 the yields of carbonization products, table 12 the physical and chemical properties of the gas, tables 13 and 14 the physical properties of the coke, and table 15 the analysis of tar distillate and light oil. Expanding properties and deterioration on oxidation were also determined.

The Lower Banner coal from Russell County, Va., (coal 91) yielded less coke and more byproducts than sample 58 from Keen Mountain mine, Buchanan County, Va. The 900° C. yields from coals 91 and 58 were, respectively: Coke, 70.3 and 79.2 percent; gas, 11,000 and 10,150 cubic feet per ton; tar, 6.9 and 3.2 percent; light oil, 3.21 and 1.92 gallons per ton; liquor, 4.7 and 4.2 percent; and ammonium sulfate, 24.2 and 15.2 pounds per ton. These differences in yields accord with the rank of the samples, for the latter (No. 91) ranks as high-volatile A, whereas the former (No. 58) was a medium-volatile coal. Variations in the yields caused by changing the carbonizing temperature were normal for high-volatile A coals. The minimum yield of coke (69.8 percent) and the maximum yields of ammonium sulfate (28.8 pounds per ton) were obtained at 800° and 700° C., respectively. The 900° C. coke was slightly weaker than the average coke from 25 high-volatile coals, as indicated by the following comparison of strength-test indexes of cokes from Lower Banner and average high-volatile A coal, respectively: 1-1/2-inch shatter, 67.3 and 70.4; 1-inch tumbler, 48.1 and 50.8; 1/4-inch tumbler, 66.7 and 72.2. The heating value of the gas from Lower Banner coal was high; the 900° C. gas had heating values of 605 B.t.u. per cubic foot and 3,330 B.t.u. per pound of coal. This gas contained a moderate proportion (210 grains per 100 cubic feet) of hydrogen sulfide. The tar contained only 2.7 percent acids, or about half the proportion generally found in high-volatile A coals; this exception may be related to the low oxygen content (6.5 percent on the as-received basis) of Lower Banner coal. The 900° C. light oil contained 76.2 percent benzene, which is higher than average.

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TABLE 10. - Analyses of coals, as-received basis[1]

Coal 28 - Pittsburgh bed, Warden mine, Allegheny County, Pa.
Coal 72 - Thick Freeport bed, Harmar mine, Harmarville, Allegheny County, Pa.
Coal 91 - Lower Banner bed, No. 56 mine, Dante, Russell County, Va.
Coal 291 - Thick Freeport bed, Indianola mine, Allegheny County, Pa.
Coal 292 - Thick Freeport bed, Russellton mine, Allegheny County, Pa.
Coal 293 - Pittsburgh bed, Crescent mine, Washington County, Pa.
Coal 294 - Pittsburgh bed, Clyde mine, Washington County, Pa.
Coal 295 - Elkhorn bed, Republic Kentucky mine, Pike County, Ky.
Coal 312 - America bed, Virginia mine, Jefferson County, Ala.
Coal 308 - Mary Lee bed, Sayreton No. 2 mine, Sayreton, Jefferson County, Ala.
Coal 309 - Mary Lee bed, Sayreton Nos. 1 and 2 mines, Sayreton, Jefferson County, Ala.
Coal 313 - Mary Lee bed, Sayre mine, Jefferson County, Ala.
Coal 301 - Pocahontas No. 3 bed, Keystone mine, McDowell County, W. Va.
Coal 302 - Illinois No. 6 bed, Old Ben No. 9 mine, West Frankfort, Franklin County, Ill.
Coal 303 - Elkhorn No. 3 bed, Weeksbury No. 1 mine, Weeksbury, Floyd County, Ky.
Coal 310 - Sample No. 1, Argentine asfaltita.
Coal a310 - Sample No. 2, Argentine asfaltita.
Coal b310 - Sample No. 3, Argentine asfaltita.
Coal 311 - No. 9 bed, Santa Maria mine, Naricual Coal Basin, Venezuela, South America.

Coal No.	Dry, mineral-matter-free, fixed carbon, percent	Proximate, percent			Ultimate, percent						Air drying loss, percent	Heating value, B.t.u. per lb.	Softening temperature of ash, °F.	Real specific gravity[2]	Carbon dioxide[3]
		Moisture	Volatile matter	Fixed carbon	Ash	Hydrogen	Carbon	Nitrogen	Oxygen	Sulfur					
28	62.6	1.8	35.1	57.7	5.4	5.3	79.0	1.6	7.8	0.9	0.7	14,140	2,740	-	0.27
72	61.7	2.8	35.3	55.6	6.3	5.4	77.4	1.5	8.2	1.2	1.3	13,810	2,300	1.33	.33
91	62.1	1.9	35.2	56.6	6.3	5.3	79.5	1.6	6.5	.8	1.1	14,170	2,340	1.32	1.65
291	61.1	2.4	35.5	54.4	7.7	5.3	76.2	1.5	8.1	1.2	1.1	13,660	2,500	1.35	.24
292	61.6	2.7	34.6	53.8	8.9	5.3	74.9	1.5	8.2	1.2	.9	13,400	2,500	1.36	.15
293	62.3	3.3	34.2	55.0	7.5	5.4	76.0	1.5	8.6	1.0	1.9	13,550	2,720	1.34	.06
294	61.0	3.1	35.7	54.6	6.6	5.5	76.4	1.6	8.6	1.3	1.7	13,650	2,500	1.34	.23
295	64.1	4.0	31.7	54.8	9.5	5.2	73.7	1.5	9.5	.6	2.7	13,170	2,660	1.36	.01
312	75.8	7.0	21.0	62.5	9.5	5.0	74.3	1.4	8.9	.9	6.3	13,060	2,730	1.37	.37
308	69.2	5.0	27.1	58.7	9.2	5.1	74.8	1.5	8.7	.7	4.4	13,270	2,910+	1.36	.26
309	68.8	7.4	26.7	56.6	9.3	5.2	72.5	1.5	10.8	.7	6.8	12,860	2,840	1.36	.34
313	67.0	6.7	28.0	54.4	10.9	5.3	71.0	1.6	10.3	.9	5.7	12,570	2,780	1.38	.10
301	82.2	1.6	16.9	74.4	7.1	4.3	82.4	1.2	4.4	.6	.9	14,290	2,340	1.37	.74
302	59.8	8.3	33.7	48.5	9.5	5.3	67.0	1.5	15.6	1.1	4.4	11,940	2,470	1.40	.23
303	59.9	3.3	38.1	56.4	2.2	5.6	80.3	1.5	9.9	.5	1.1	14,290	2,150	1.30	.33
310	68.4	1.0	30.0	63.1	5.9	5.9	81.6	1.7	2.8	2.1	.3	14,990	2,190	1.30	1.32
a310	54.2	1.6	43.7	51.0	3.7	7.2	82.3	1.6	2.9	2.3	1.1	15,690	2,590	1.21	1.13
b310	53.5	.9	44.8	51.0	3.3	7.3	83.4	1.6	2.1	2.3	.4	15,990	2,210	1.21	.97
311	54.8	3.6	42.6	51.7	2.1	6.0	76.2	2.3	12.9	.5	1.5	13,730	1,990	1.28	.27

1/ Analyses made under supervision of H. M. Cooper, chemist, Bureau of Mines.
2/ Air-dried coal.
3/ Determined on air-dried coal; total carbon not corrected for this carbon.

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TABLE 11. - Yields of carbonization products at 900° C., as-carbonized basis

Coal 28 - Pittsburgh bed, Warden mine, Allegheny County, Pa.
Coal 72 - Thick Freeport bed, Harmar mine, Harmarville, Allegheny County, Pa.
Coal 91 - Lower Banner bed, No. 56 mine, Dante, Russell County, Va.
Coal 291 - Thick Freeport bed, Indianola mine, Allegheny County, Pa.
Coal 292 - Thick Freeport bed, Russellton mine, Allegheny County, Pa.
Coal 293 - Pittsburgh bed, Crescent mine, Washington County, Pa.
Coal 294 - Pittsburgh bed, Clyde mine, Washington County, Pa.
Coal 295 - Elkhorn bed, Republic Kentucky mine, Pike County, Ky.
Coal 312 - America bed, Virginia mine, Jefferson County, Ala.
Coal 308 - Mary Lee bed, Sayreton No. 2 mine, Sayreton, Jefferson County, Ala.
Coal 309 - Mary Lee bed, Sayreton Nos. 1 and 2 mines, Sayreton, Jefferson County, Ala.
Coal 313 - Mary Lee bed, Sayre mine, Jefferson County, Ala.
Coal 301 - Pocahontas No. 3 bed, Keystone mine, McDowell County, W. Va.
Coal 302 - Illinois No. 6 bed, Old Ben No. 9 mine, West Frankfort, Franklin County, Ill.
Coal 303 - Elkhorn No. 3 bed, Weeksbury No. 1 mine, Weeksbury, Floyd County, Ky.
Coal 310 - Sample No. 1, Argentine asfaltita.
Coal 310A - 50 percent sample No. 1 and 50 percent sample No. 2 (a310).
Coal 310B - 50 percent sample No. 1 and 50 percent sample No. 3 (b310).
Coal 311 - No. 9 bed, Santa Maria mine, Naricual Coal Basin, Venezuela, South America.
Coal 311A - 90 percent coal 311 and 10 percent Venezuelan pitch.

Coal No.	Retort diameter, inches	Yields, percent by weight of coal[1]							Yields per ton of coal[1]				
		Coke	Gas	Tar	Light oil	Free ammonia	Liquor	Total	Gas, cubic feet	Tar, gallons	Light oil, gal. in gas	Light oil, gal. Tar to 170° C.	$(NH_4)_2SO_4$, pounds
28	18	68.8	14.5	7.3	1.01	0.110	6.4	98.1	10,650	14.9	2.79	0.75	19.0
72	18	67.8	15.2	7.3	1.22	.158	6.7	98.4	10,500	15.1	3.39	.69	19.7
91	18	70.3	16.0	6.9	1.17	.188	4.7	99.3	11,000	13.8	3.21	.71	24.2
291	18	68.7	14.9	7.2	1.24	.185	6.6	98.8	10,350	14.8	3.43	.73	20.8
292	18	68.8	15.2	6.5	1.32	.179	7.0	99.0	10,300	13.1	3.62	.50	19.5
293	18	68.2	14.0	7.0	1.28	.184	8.0	98.7	10,100	14.4	3.56	.72	21.9
294	18	66.7	15.0	7.4	1.32	.197	8.2	98.8	10,450	14.9	3.63	.54	22.1
295	18	70.4	13.7	5.3	1.02	.188	7.9	98.5	9,850	10.9	2.82	.50	22.4
312	18	75.3	11.4	3.2	.66	.176	9.3	100.0	9,650	6.6	1.83	.28	22.6
308	18	72.7	12.9	5.4	.77	.171	8.3	100.2	10,050	10.9	2.14	.40	26.1
309	18	70.7	12.4	5.0	.86	.168	10.5	99.6	9,650	10.3	2.40	.49	27.6
313	18	69.9	12.8	5.4	.84	.180	10.9	100.0	9,300	11.2	2.34	.46	26.6
301	13	83.6	10.2	1.3	.42	.118	2.6	98.2	10,050	2.6	1.14	.16	10.8
302	13	63.9	14.7	5.0	.97	.120	13.4	98.1	9,750	10.3	2.66	.63	28.3
303	13	66.0	16.5	6.9	1.22	.108	7.4	98.1	10,950	14.2	3.38	.91	20.3
310	13	72.3	19.8	3.3	1.51	.220	1.7	98.8	12,550	6.9	4.13	.66	17.1
310A	13	65.6	22.4	5.7	2.14	.151	1.5	97.5	13,350	11.7	5.86	.81	11.7
310B	13	65.6	23.1	6.1	2.38	.167	1.1	98.4	13,450	12.5	6.50	.94	13.0
311	13	59.6	23.1	6.2	1.16	.126	7.9	98.1	12,550	12.9	3.22	.95	29.6
311A	10	58.9	24.2	8.2	1.76	.169	4.3	97.5	12,900	16.8	4.81	1.33	19.9

1/ Coke, tar, ammonia, and light oil are reported moisture-free; gas is reported as stripped of light oil and saturated with water vapor at 60° F. and under a pressure of 30 inches of mercury.

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TABLE 12. - Physical and chemical properties of gas at 900° C.

Coal 28 - Pittsburgh bed, Warden mine, Allegheny County, Pa.
Coal 72 - Thick Freeport bed, Harmar mine, Harmarville, Allegheny County, Pa.
Coal 91 - Lower Banner bed, No. 56 mine, Dante, Russell County, Va.
Coal 291 - Thick Freeport bed, Indianola mine, Allegheny County, Pa.
Coal 292 - Thick Freeport bed, Russellton mine, Allegheny County, Pa.
Coal 293 - Pittsburgh bed, Crescent mine, Washington County, Pa.
Coal 294 - Pittsburgh bed, Clyde mine, Washington County, Pa.
Coal 295 - Elkhorn bed, Republic Kentucky mine, Pike County, Ky.
Coal 312 - America bed, Virginia mine, Jefferson County, Ala.
Coal 308 - Mary Lee bed, Sayreton No. 2 mine, Sayreton, Jefferson County, Ala.
Coal 309 - Mary Lee bed, Sayreton Nos. 1 and 2 mines, Sayreton, Jefferson County, Ala.
Coal 313 - Mary Lee bed, Sayre mine, Jefferson County, Ala.
Coal 301 - Pocahontas No. 3 bed, Keystone mine, McDowell County, W. Va.
Coal 302 - Illinois No. 6 bed, Old Ben No. 9 mine, West Frankfort, Franklin County, Ill.
Coal 303 - Elkhorn No. 3 bed, Weeksbury No. 1 mine, Weeksbury, Floyd County, Ky.
Coal 310 - Sample No. 1, Argentine asfaltita.
Coal 310A - 50 percent sample No. 1 and 50 percent sample No. 2 (a310).
Coal 310B - 50 percent sample No. 1 and 50 percent sample No. 3 (b310).
Coal 311 - No. 9 bed, Santa Maria mine, Naricual Coal Basin, Venezuela, South America.
Coal 311A - 90 percent 311 and 10 percent Venezuelan pitch.

Coal No.	Retort diameter, inches	Specific gravity	Gross heating value 1/ B.t.u. per cubic foot (Determined)	Gross heating value 1/ B.t.u. per cubic foot (Calculated)	Gross heating value 1/ B.t.u. per pound of coal	H_2S, grains per 100 cubic feet	Composition, dry, percent by volume: CO_2	Illuminants	O_2	H_2	CO	CH_4	C_2H_6	N_2
28	18	0.362	607	595	3,230	200	1.0	4.4	0.3	53.7	6.5	31.9	1.0	1.2
72	18	.385	617	608	3,240	320	2.0	4.9	.2	51.8	6.3	32.3	1.5	1.0
91	18	.385	605	585	3,330	210	1.9	4.6	.4	53.1	6.0	32.8	.0	1.2
291	18	.384	615	591	3,180	440	1.9	4.9	.3	52.1	6.0	32.9	.2	1.7
292	18	.392	606	599	3,120	450	2.2	5.4	.4	51.4	6.1	33.4	.0	1.1
293	18	.369	598	576	3,020	400	2.0	4.7	.4	54.0	6.6	31.1	.0	1.2
294	18	.382	586	582	3,060	500	2.1	4.7	.4	52.9	6.7	32.1	.0	1.1
295	18	.370	595	587	2,930	250	1.8	4.4	.3	53.2	6.2	31.8	.8	1.5
312	18	.313	559	541	2,700	190	1.3	2.4	.4	61.0	3.7	29.8	.0	1.4
308	18	.340	572	567	2,870	180	1.3	3.4	.4	57.3	4.6	31.9	.0	1.1
309	18	.343	572	560	2,760	190	1.4	3.4	.4	58.1	4.7	30.9	.0	1.1
313	18	.365	591	566	2,750	310	1.8	3.2	.2	55.0	5.9	32.5	.0	1.4
301	13	.269	481	480	2,420	80	1.2	1.4	.4	69.1	4.1	22.7	.0	1.1
302	13	.399	545	537	2,660	430	3.6	3.6	.4	53.3	9.9	28.0	.1	1.1
303	13	.398	601	588	3,290	190	2.4	5.2	.4	51.1	7.8	31.7	.3	1.1
310	13	.420	710	689	4,450	110	1.1	9.5	.4	46.2	2.8	38.8	.0	1.2
310A	13	.446	746	718	4,980	290	1.3	9.2	.4	41.5	2.9	43.7	.0	1.0
310B	13	.455	729	763	4,900	330	1.4	10.4	.4	41.2	2.6	42.6	.3	1.1
311	13	.490	614	600	3,850	120	5.2	6.9	.4	44.3	10.0	32.2	.0	1.0
311A	10	.500	679	661	4,380	180	3.9	9.3	.4	41.0	8.1	36.3	.0	1.0

1/ Stripped of light oil and saturated with water vapor at 60° F. and under a pressure equivalent to 30 inches of mercury.

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TABLE 13. - Physical properties of coke at 900° C. (Bureau of Mines method)

Coal 28 - Pittsburgh bed, Warden mine, Allegheny County, Pa.
Coal 72 - Thick Freeport bed, Harmar mine, Harmarville, Allegheny County, Pa.
Coal 91 - Lower Banner bed, No. 56 mine, Dante, Russell County, Va.
Coal 291 - Thick Freeport bed, Indianola mine, Allegheny County, Pa.
Coal 292 - Thick Freeport bed, Russellton mine, Allegheny County, Pa.
Coal 293 - Pittsburgh bed, Crescent mine, Washington County, Pa.
Coal 294 - Pittsburgh bed, Clyde mine, Washington County, Pa.
Coal 295 - Elkhorn bed, Republic Kentucky mine, Pike County, Ky.
Coal 312 - America bed, Virginia mine, Jefferson County, Ala.
Coal 308 - Mary Lee bed, Sayreton No. 2 mine, Sayreton, Jefferson County, Ala.
Coal 309 - Mary Lee bed, Sayreton Nos. 1 and 2 mines, Sayreton, Jefferson County, Ala.
Coal 313 - Mary Lee bed, Sayre mine, Jefferson County, Ala.
Coal 301 - Pocahontas No. 3 bed, Keystone mine, McDowell County, W. Va.
Coal 302 - Illinois No. 6 bed, Old Ben No. 9 mine, West Frankfort, Franklin County, Ill.
Coal 303 - Elkhorn No. 3 bed, Weeksbury No. 1 mine, Weeksbury, Floyd County, Ky.

Coal No.	Retort diameter, inches	True specific gravity	Apparent specific gravity	Cells, percent	Shatter test, cumulative percent upon-				Tumbler test, cumulative percent upon-			
					2-inch screen	1-1/2-inch screen	1-inch screen	1/4-inch screen	2-inch screen	1-1/2-inch screen	1-inch screen	1/4-inch screen
28	18	1.85	0.88	52.5	33.1	68.0	93.2	98.6	1.8	20.4	55.2	71.5
72	18	1.87	.82	56.1	42.3	76.5	91.9	98.0	.1	18.4	52.6	71.8
91	18	1.89	.88	53.4	34.1	67.3	91.7	97.9	.6	11.1	48.1	66.7
291	18	1.91	.89	53.4	35.3	66.3	89.2	97.9	.0	13.2	49.2	70.7
292	18	1.92	.90	53.1	41.4	68.9	88.2	97.8	.3	14.4	49.1	72.8
293	18	1.91	.85	55.5	33.2	70.6	92.6	97.9	1.6	20.2	53.9	70.2
294	18	1.92	.83	56.8	28.4	68.0	92.0	98.1	.0	16.5	54.4	71.9
295	18	1.92	.82	57.3	59.9	83.4	92.8	97.9	8.7	35.9	56.5	70.7
312	18	1.94	.87	55.2	64.8	86.7	95.0	98.3	9.3	36.8	58.2	67.0
308	18	1.93	.85	56.0	61.6	87.4	95.9	98.4	13.0	44.4	64.7	70.4
309	18	1.92	.85	55.7	55.9	87.4	96.4	98.5	9.4	40.1	63.2	68.7
313	18	1.96	.82	58.2	54.9	85.1	95.8	98.4	9.0	36.9	61.8	68.9
301	13	1.91	.91	52.4	49.2	80.2	95.2	98.8	.0	15.5	54.6	71.1
302	13	1.95	.75	61.5	22.1	49.7	78.6	97.0	.0	1.1	25.8	68.7
303	13	1.87	.74	60.4	11.3	40.2	79.6	97.9	.0	.8	30.8	74.8

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TABLE 14. - Physical properties of coke at 900° C. (Columbia Steel Co. methods)

Coal 310 - Sample No. 1, Argentine asfaltita.
Coal 310A - 50 percent sample No. 1 and 50 percent sample No. 2 (a310).
Coal 310B - 50 percent sample No. 1 and 50 percent sample No. 3 (b310).
Coal 311 - No. 9 bed, Santa Maria mine, Naricual Coal Basin, Venezuela, South America.
Coal 311A - 90 percent coal 311 and 10 percent Venezuelan pitch.

Coal No.	Retort diameter, inches	True specific gravity	Apparent specific gravity	Cells, percent	Shatter test, cumulative percent upon-				Tumbler test, cumulative percent upon-			
					1-1/2-inch screen	1-inch screen	3/4-inch screen	1/2-inch screen	1-1/2-inch screen	1-inch screen	3/4-inch screen	1/2-inch screen
310	13	1.88	-	-	-	-	-	-	0.0	1.4	11.6	31.4
310A	13	1.90	0.84	55.8	27.8	53.8	66.1	77.2	.0	17.8	30.7	43.7
310B	13	1.90	.69	63.7	33.8	60.1	69.1	79.1	1.1	19.6	32.1	43.5
311	13	1.88	.77	59.0	-	-	-	-	.0	16.2	40.6	54.4
311A	10	1.89	.80	57.7	-	-	-	-	.0	17.4	40.6	60.0

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TABLE 15. - Analysis of tar distillate and light oil at 900° C.

Coal 28 - Pittsburgh bed, Warden mine, Allegheny County, Pa.
Coal 72 - Thick Freeport bed, Harmar mine, Harmarville, Allegheny County, Pa.
Coal 91 - Lower Banner bed, No. 56 mine, Dante, Russell County, Va.
Coal 291 - Thick Freeport bed, Indianola mine, Allegheny County, Pa.
Coal 292 - Thick Freeport bed, Russellton mine, Allegheny County, Pa.
Coal 293 - Pittsburgh bed, Crescent mine, Washington County, Pa.
Coal 294 - Pittsburgh bed, Clyde mine, Washington County, Pa.
Coal 295 - Elkhorn bed, Republic Kentucky mine, Pike County, Ky.
Coal 312 - America bed, Virginia mine, Jefferson County, Ala.
Coal 308 - Mary Lee bed, Sayreton No. 2 mine, Sayreton, Jefferson County, Ala.
Coal 309 - Mary Lee bed, Sayreton Nos. 1 and 2 mines, Sayreton, Jefferson County, Ala.
Coal 313 - Mary Lee bed, Sayre mine, Jefferson County, Ala.
Coal 301 - Pocahontas No. 3 bed, Keystone mine, McDowell County, W. Va.
Coal 302 - Illinois No. 6 bed, Old Ben No. 9 mine, West Frankfort, Franklin County, Ill.
Coal 303 - Elkhorn No. 3 bed, Weeksbury No. 1 mine, Weeksbury, Floyd County, Ky.
Coal 310 - Sample No. 1, Argentine asfaltita.
Coal 310A - 50 percent sample No. 1 and 50 percent sample No. 2 (a310).
Coal 310B - 50 percent sample No. 1 and 50 percent sample No. 3 (b310).
Coal 311 - No. 9 bed, Santa Maria mine, Naricual Coal Basin, Venezuela, South America.
Coal 311A - 90 percent coal 311 and 10 percent Venezuelan pitch.

Coal No.	Retort diameter, inches	Distillate, percent by volume of dry tar			Neutral tar oil, percent by volume			Refined light oil from gas, percent by volume				Olefins in crude light oil from gas, percent by volume
		Acids	Bases	Neutral oils	Olefins	Aromatics	Paraffins and naphthenes	Benzene	Toluene	Paraffins	Solvent naphtha	
28	18	4.8	1.6	26.2	12.3	84.6	3.1	72.7	19.5	2.4	5.4	8.4
72	18	7.3	1.8	28.1	8.8	86.2	5.0	63.0	20.7	6.2	10.1	13.0
91	18	2.7	1.5	21.7	11.9	86.6	1.5	76.2	17.6	3.2	3.0	7.6
291	18	6.0	1.7	27.2	9.6	87.0	3.4	65.0	21.8	5.5	7.7	11.4
292	18	3.7	1.8	22.6	10.3	89.0	.7	72.8	19.7	2.7	4.8	8.7
293	18	6.2	2.1	27.7	9.5	87.2	3.3	65.0	20.3	7.5	7.3	11.1
294	18	6.8	1.9	22.8	9.9	88.7	1.4	67.8	20.7	4.6	6.9	9.7
295	18	6.4	2.1	28.5	9.3	87.5	3.2	65.7	21.2	5.7	7.4	12.0
312	18	2.4	1.3	22.6	8.8	77.1	14.1	69.0	18.4	5.1	7.5	10.6
308	18	3.2	1.8	21.8	11.3	78.2	10.5	69.8	19.0	5.3	5.9	12.6
309	18	3.4	1.0	22.4	10.3	79.4	10.3	71.7	18.1	5.1	5.1	12.0
313	18	6.5	2.0	27.4	10.0	81.0	9.0	66.4	19.2	7.9	6.5	12.4
301	13	1.3	1.3	11.3	14.1	85.2	.7	69.7	21.1	2.4	6.8	11.8
302	13	7.2	3.1	27.5	10.6	88.5	.9	72.7	22.0	1.6	3.7	11.2
303	13	6.6	1.5	27.8	12.9	83.9	3.2	72.0	18.6	5.4	4.0	11.3
310	13	.5	1.7	27.6	12.5	84.4	3.1	79.3	17.1	1.2	2.4	9.2
310A	13	.7	.9	20.8	16.0	83.2	.8	84.2	11.7	2.4	1.7	8.8
310B	13	.5	.9	23.9	7.9	90.7	1.4	80.0	16.6	1.7	1.7	8.0
311	13	6.0	3.5	29.5	12.5	85.3	2.2	72.5	18.9	3.8	4.8	13.7
311A	10	2.9	2.7	26.9	14.1	84.6	1.3	80.1	15.1	3.1	1.7	9.0

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The principal high-volatile coals for metallurgical coke are obtained from a number of sources. The Thick Freeport and Pittsburgh beds in Pennsylvania and the Elkhorn bed in Kentucky furnishes large quantities. Mixtures of medium- to high-volatile Mary Lee and America coals are used in Alabama. Low-volatile coals for blending with the Freeport, Pittsburgh, and Elkhorn are purchased on the open market, and it has not been possible to obtain low-volatile coal of uniform quality for the various plants using these coals. Recovery of byproducts from several plants has differed considerably and it was desired to know how much of the differences in yields could be charged to differences in coals used. Accordingly, it was decided to test the coals, in cooperation with the Republic Steel Corp., by the BM-AGA method and thus obtain a reliable basis for comparing them. Blending with miscellaneous low-volatile coals at byproduct-oven plants complicated the comparison, but only small amounts of these coals are used, and in some cases BM-AGA results are available on them.

Nine coals were carbonized in duplicate in the 18-inch retort at 800°, 900°, and 1,000° C. The usual differences in yields of products were obtained on going from 800° to 1,000° C. That is, as the carbonizing temperature is raised, the yield of gas increases and that of tar decreases; the yield of coke at 1,000° C. is usually slightly higher than that at 900° C.

As two Thick Freeport- and two Pittsburgh-bed coals from mines only a short distance apart in each case were tested, the yields were calculated to the ash- and moisture-free basis to bring out differences in carbonizing properties of the coal substance. Results for the Harmar mine (coal 72), which is also a Thick Freeport bed coal from Allegheny County, and for Pittsburgh bed (coal 28), Warden mine, coal are also included. The yields from the Indianola and Harmar mine coals (table 16) are almost identical; the Russellton coal yields more gas, less tar, and slightly more light oil. It is believed that this increase in light oil is large enough to be considered significant.

Clyde coal gave a lower yield of coke and higher yields of tar and gas than the other two Pittsburgh-bed coals; the yields from Crescent and Warden coals were very similar. The yield of light oil from the latter was low, but this coal was tested more than 15 years ago when automatic cooling for the light-oil scrubbers had not been provided. Probably some of the light oil from the Warden coal was not removed from the gas. The figures for heat-in-gas per pound of coal carbonized indicate that this is true; they are 3,230 B.t.u. for Warden and 3,020 for Crescent. Elkhorn coal yielded more coke and less gas, tar, and light oil than Thick Freeport and Pittsburgh coals.

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TABLE 16. - Yields of carbonization products in the 18-inch retort at 900° C., ash- and moisture-free basis

Coal No.	Coal bed	Mine	County	Percent of coal substance							Yields, per ton of coal			
				Coke	Gas	Tar	Light oil	Free ammonia	Liquor	Total	Gas, cubic feet	Tar, gallons	Light oil, gallons	$(NH_4)_2SO_4$, pounds
291	Thick Freeport	Indianola	Allegheny	67.7	16.0	8.0	1.38	0.206	4.7	98.6	11,500	16.5	3.8	23.1
292	do.	Russellton	do.	67.7	17.2	7.4	1.49	.202	4.9	98.9	11,650	14.8	4.1	22.1
72	do.	Harmar	do.	68.0	16.7	8.0	1.34	.170	4.3	98.5	11,550	16.6	3.7	21.7
293	Pittsburgh	Crescent	Washington	68.1	15.7	7.8	1.43	.210	5.3	98.5	11,320	16.1	4.0	24.6
294	do.	Clyde	do.	66.3	16.6	8.2	1.46	.210	5.7	98.5	11,570	16.5	4.0	24.5
28	do.	Warden	Allegheny	68.5	15.6	7.9	1.09	.120	5.0	98.2	11,470	16.1	3.0	20.5

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Generally, the properties of the cokes correlate with rank of coal. The five high-volatile A coals representing the Thick Freeport, Pittsburgh, and Elkhorn beds coked less strongly than the higher-ranking Alabama coals. The 900° C. coke from Elkhorn coal was outstanding for the coal rank; the 1-½-inch shatter and 1-inch tumbler indexes of this coke were 83.4 and 56.5, respectively, whereas the corresponding averages for 25 high-volatile A coals were 70.4 and 50.8, respectively. The three Mary Lee coals ranked low in the medium-volatile classification or very high in the high-volatile A group, and America coal ranked high in the medium-volatile group. The cokes from these coals were definitely stronger than the cokes from the five high-volatile A coals, if judged by the 1-½-inch shatter and 1-inch tumbler indexes. The 1/4-inch tumbler indexes are not outstanding for either coal group. Although America coal ranked highest, its coking power approximately equaled that of Elkhorn coal. Previous investigations of coals from these beds indicate that they yield metallurgical coke if blended with coals of suitable rank and composition.

When the BM-AGA work was started, it appeared that crushing the coal for carbonization to pass ¼-inch corresponded to the average coke-oven practice; accordingly, this size was chosen for the testing, and coals were stage-crushed so that there was a minimum of fines. Now, however, finer crushing is practiced at coke plants; most plants crush to 0- to 1/8-inch with about 70 percent through 1/8-inch, particularly where it is necessary to blend several coals. The quality of the coke is believed improved thereby. It seems obvious that the BM-AGA preparation should be changed to correspond more closely with industrial practice, and tests of the effect of fine and coarse crushing on the physical properties of the coke have been made from time to time. To obtain more data on this effect, the five high-volatile coals were tested at 900° C. using both the 0- to ¼-inch and 0- and 1/8-inch sizes. The results are given in table 17. The improvement in the 1-inch tumbler index of the coke is definite, and the ¼-inch index generally is raised by the reduction in size. The 1-½-inch shatter index shows appreciable improvement for the Thick Freeport coals, but not for the Pittsburgh and Elkhorn coals. If the comparison is made on the basis of the 1-inch index, improvement is shown except for the Crescent coal.

The physical and chemical properties of the 900° C. gases shown in table 12 indicate that none of the North American coals tested rank high as gas coals. The heating values were highest for Thick Freeport and Pittsburgh coals; however, these ranged from 3,020 to 3,180 B.t.u. per pound of coal, whereas the average value for 39 high-volatile A coals is 3,220 B.t.u. per pound. The gases from the other coals had heating values lower than 3,000 B.t.u. per pound. The content of hydrogen sulfide in the 900° C. gases ranged from 180 to 500 grains per 100 cubic feet, the northern coals having the highest proportions. However, the samples of America and Mary Lee coals had been washed, which probably lowered the sulfur content of the coal appreciably.

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TABLE 17. - Effect of fine crushing of coal on physical properties of coke

Coal	Crushing	Shatter index, cumulative percent upon-		Tumbler index, cumulative percent upon-	
		1-1/2-inch screen	1-inch screen	1-inch screen	1/4-inch screen
Indianola	0- to 1/4-inch	66.3	89.2	49.2	70.7
Do.	0- to 1/8-inch	74.1	91.5	58.6	74.5
Russellton	0- to 1/4-inch	68.9	88.2	49.1	72.8
Do.	0- to 1/8-inch	77.0	92.3	56.2	73.0
Crescent	0- to 1/4-inch	70.6	92.6	53.9	70.2
Do.	0- to 1/8-inch	70.6	92.5	55.8	70.7
Clyde	0- to 1/4-inch	68.0	92.0	54.4	71.9
Do.	0- to 1/8-inch	66.1	93.1	58.4	71.8
Elkhorn	0- to 1/4-inch	83.4	92.8	56.5	70.7
Do.	0- to 1/8-inch	79.5	93.4	62.9	75.3

The properties of the tar and light oil do not correlate closely with coal rank. The proportion of acids in the tar distillate is lowest (2.4 percent) for the highest-ranking coal (America), but the percentage for Sayre mine, Mary Lee Bed, coal is 6.5; and this coal ranks higher than Russellton mine, Thick Freeport-bed coal, for which the percentage is only 3.7. The percentages of paraffins and naphthenes in the neutral oils was definitely higher for the higher-ranking coals from Alabama. The proportions of benzene and toluene in the light oils ranged from 65.0 to 72.8 and from 18.1 to 21.8, respectively, with virtually no relation to rank of coal.

Argentine Asfaltita

Three samples of Argentine asfaltita, each weighing about 300 pounds, were received for carbonization tests made in the 13-inch BM-AGA retort at 900° C. The samples were marked Nos. 1, 2, and 3 and were distinguished by the amount of asphaltic material soluble in carbon disulfide; extractable matter was 8.4, 49.7, and 48.8 for samples 1, 2, and 3, respectively. Abraham's "Asphalts and Allied Substances" calls the material asphaltite, or more specifically, "grahamite." The moisture contents were 1.0, 1.6, and 0.9 percent; the ash, 5.9, 3.7, and 3.3 percent; the sulfur 2.1, 2.3, and 2.3 percent; and heating values, 14,990, 15,690, and 15,990 B.t.u. respectively, for the three samples. Sample 1 could be carbonized 100 percent, but samples 2 and 3 swelled out of the retorts and could be carbonized only by mixing with equal parts of sample 1.

The cokes from asfaltita resembled those from Utah coals in structure, but they were much weaker than those from the best Utah coals. It is believed, however, that they are strong enough to serve as domestic fuel. Because of the high asphaltic content, the yield and quality of the gas was

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very high; the heating value per cubic foot ranged from 710 to 746 B.t.u., and the heat in gas per pound of material ranged from 4,450 to 4,980 B.t.u. The tar and light oil were highly aromatic. Aromatic compounds in tar were concentrated in naphthalene and anthracene salts, and in the light oil they appeared largely as benzene. The highest yield of pure benzene obtained was about five gallons per ton.

Venezuelan Coal

A 400-pound sample of Naricual Basin coal, Venezuela, contained 3.6 percent moisture, 2.1 percent ash, and 0.5 percent sulfur. It contained 34.8 percent fixed carbon on the dry, mineral-matter-free basis and had a heating value of 14,060 on the moist, mineral-matter-free basis; therefore, it ranks as high-volatile A coal. It was known to be deficient in coking properties, and the problem was to determine if metallurgical-quality coke could be produced from blends of this coal and asphaltic oil-still residue. BM-AGA tests at 900° C. indicated that blending with 10 percent still residue improved the coke, although it was still inferior to that from Lower Sunnyside (Utah) coal. Further experiments, using larger proportions of residue, may beneficiate the coke more, but richer mixtures are difficult to prepare and carbonize; they give excessive amounts of spongy coke at the center of the charge.

Naricual coal yielded 59.6 percent coke, 23.1 percent gas (equivalent to 12,550 cubic feet per ton), 12.9 gallons of tar, 4.17 gallons of light oil, and 29.6 pounds of ammonium sulfate per ton. The heating values of the gas (614 B.t.u. per cubic foot and 3,850 B.t.u. per pound of coal) were high. The composition of the tar and light oil was similar to that from domestic high-volatile coals. Blending with asphaltic residue raised the heating values of the gas significantly and increased the proportion of benzene in the light oil.

Expansion-Test Investigations

Figure 35 shows a vertical section of the small vertical oven, and figure 36 is a photograph showing the oven open. The coking chamber is 12 inches long and 5 inches wide and holds about 17 pounds of coal when filled to a depth of 9-½ inches. The bottom and ends are fire brick, and the heated walls are silicon carbide plates. Each wall is heated by nine horizontal coils of 13-gage nickel-chromium wire, with resistance adjusted to give uniform heating. The maximum variation in the temperature across the walls is 25° C., a large part of which occurs near the edges of the walls.

A large number of tests have been made over a period of 3 years by several operators with various adjustments of test conditions in the hope that a schedule could be worked out that would produce uniform results. Results in table 18 are typical of the unsatisfactory performance of the oven.

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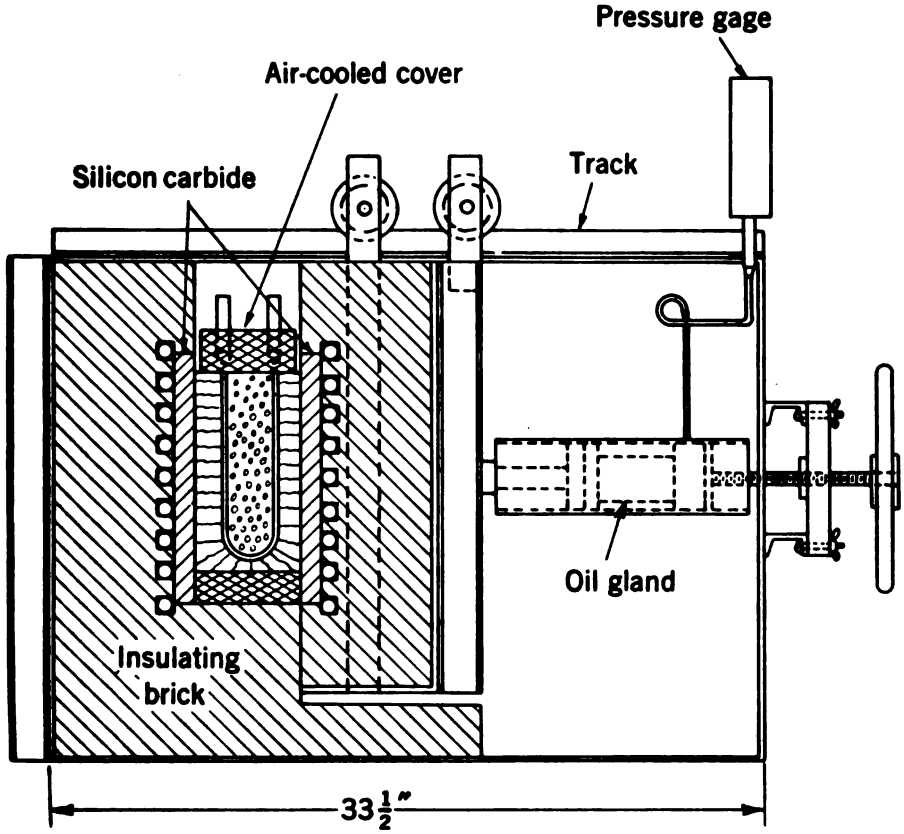

Figure 35. - Small, vertical oven.

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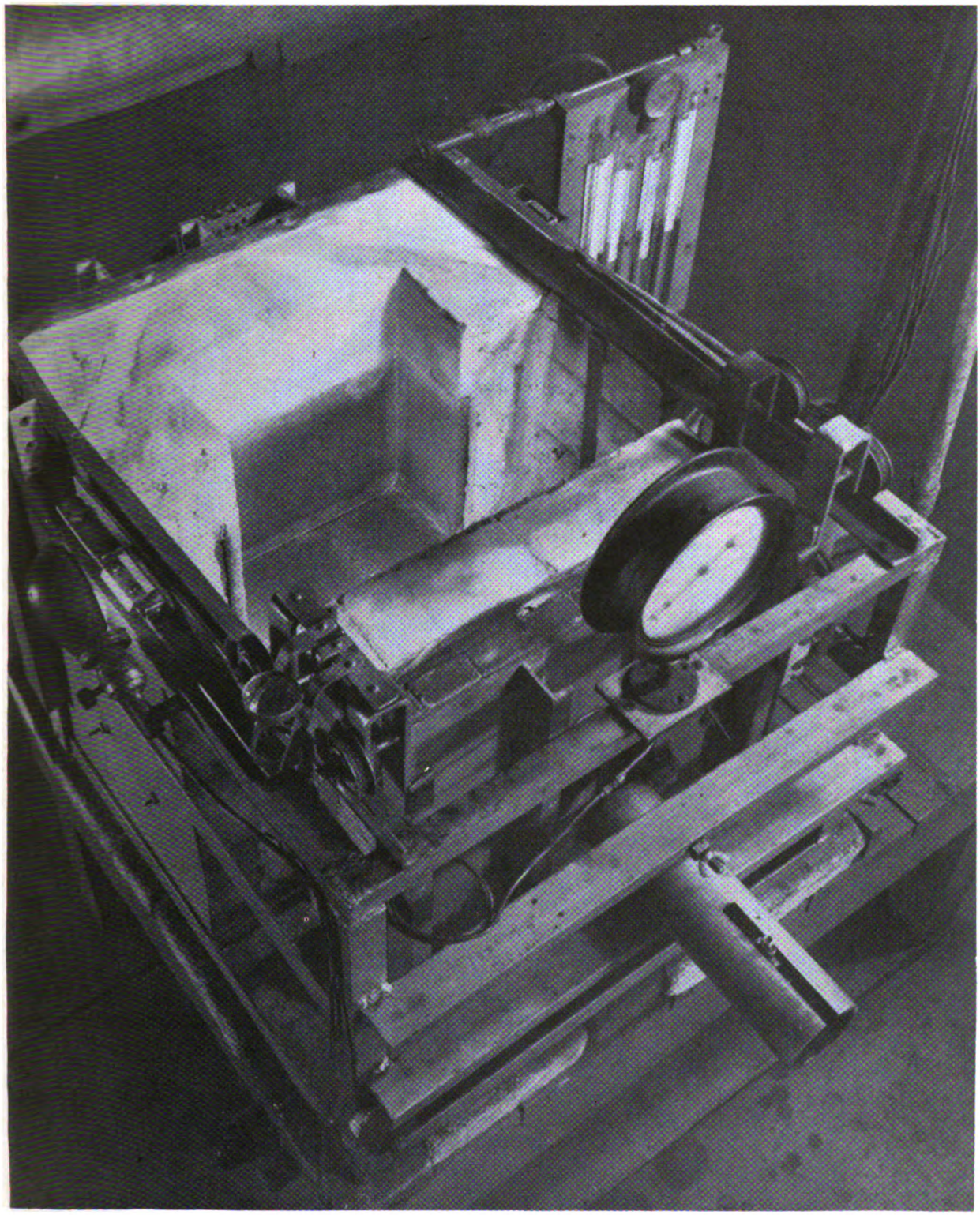

Figure 36. - Small, vertical oven, showing interior.

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TABLE 18. - Agreement of test results from small vertical oven

Coal 89 - 100 percent Pocahontas No. 6 bed
Coal 82D - 35 percent Eagle bed, 35 percent Powellton bed, and 30 percent Pocahontas No. 3 bed
Coal 235 - From coal-charging car at coke oven at Rosita, Coahuila, Mexico
Coal 238B - 70 percent Powellton bed and 30 percent Pocahontas No. 3 bed

Coal No.	Test No.	Bulk density, pounds per cubic foot	Maximum pressure, pounds per square inch		Deviation of wall pressure from the average
			Gas	Wall	
		Free top.	2,000°-2,200° F. schedule		
89	SV-14	49.0	14.0	7.7	+1.6
	SV-8	49.1	14.0	7.3	+1.2
	SV-15	49.2	4.2	2.1	-4.0
	SV-9	49.4	9.0	7.2	+1.1
	Average	49.2	10.3	6.1	2.0
89	SV-2	50.0	9.0	3.2	-1.6
	SV-12	50.0	8.0	3.6	-1.2
	SV-13	50.3	13.0	5.8	+1.0
	SV-10	50.3	12.0	5.8	+1.0
	SV-7	50.6	4.9	3.3	-1.5
	SV-6	50.6	15.0	7.0	+2.2
	Average	50.3	10.3	4.8	1.4
89	SV-1	51.3	8.0	8.0	+2.7
	SV-4	51.3	11.7	5.2	- .1
	SV-5	51.3	9.0	5.4	+ .1
	SV-11	51.9	5.0	2.4	-2.9
	Average	51.5	8.4	5.3	1.5
		Confined top - cooled.	2,000°-2,200° F. schedule		
82D	SV-7	48.2	6.0	3.0	-0.2
	SV-8	50.4	5.5	3.2	.0
	SV-9	50.4	2.2	3.9	+ .7
	SV-10	50.4	4.0	2.7	- .5
	SV-14	50.4	6.0	3.0	- .2
	Average	50.0	4.7	3.2	.3
235	SV-4	51.2	-	1.9	-0.5
	SV-5	51.2	-	2.3	- .1
	SV-6	51.2	-	2.9	+ .5
	SV-7	51.2	-	1.8	- .6
	SV-8	51.2	-	2.5	+ .1
	SV-9	51.2	-	2.8	+ .4
	Average	51.2	-	2.4	.4
238B	SV-1	51.2	-	8.2	+3.7
	SV-2	51.2	-	4.3	- .2
	SV-3	51.2	-	4.9	+ .4
	SV-4	51.2	-	4.6	+ .1
	SV-5	51.2	-	3.8	- .7
	SV-6	51.2	-	3.2	-1.3
	SV-7	51.2	-	2.7	-1.8
	Average	51.2	-	4.5	1.2

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Table 19 compares results on the same coals for the sole-heated, large vertical, and small vertical slot ovens. The coals expanding most in the sole-heated oven show the highest pressures in both vertical ovens, but the tests do not place the coals in the same order. The small vertical oven gives the highest pressures, except in one case, and here the maximum pressure is so small as to be almost within limits of experimental error. The gas pressure is always high in the small vertical oven, and it is erratic. This is undoubtedly a contributing factor in the high wall pressure for this oven and also in lack of agreement between duplicate results. The high ratio of thickness of the plastic layers at the center to the total thickness of the charge in the small vertical oven would favor high expansion pressures; or, stated in another way, the pressure developed at the center is less-relieved by the relatively thin layer of contracting coke. Development of high erratic gas pressures behind the plastic envelope in the small vertical oven is perhaps its more serious defect. Other factors contributing to high results in this oven should be constant under uniform test conditions, and it therefore should be possible to correlate them with those of the larger test ovens.

The sole-heated oven was used in 101 tests to determine the expanding properties of the 29 coal samples and blends described in table 20. New samples of standard high- and low-volatile coals used for blending with BM-AGA coals were received and tested in the sole-heated oven. High-volatile Pittsburgh-bed, Warden mine coal (m28), contracted 14.0 percent as compared with 13.7 percent for a sample received in 1938. This coal was oxidized in air at 100° C. for various periods and tested after each stage in the sole-heated oven. The results of these tests are given in table 21. The low-volatile sample, Pocahontas No. 3 bed, Carswell mine (coal d75), expanded 19.7 percent as compared with 21.7 and 24.4 percent for former samples.

Lower Banner-bed coal from No. 56 mine, Dante, Russell County, Va. (coal 91), contracted 25.8 percent in the sole-heated oven. The oxidized samples contracted less as oxidation progressed. This increase in expanding tendency may account for the improvement in coke quality sometimes found with slight oxidation. The 1-½-inch shatter index of the coke from this coal was not improved, but the 1-inch tumbler index was increased from 49.7 percent for the unoxidized coal to 57.9 percent for the second oxidation stage. The tumbler index decreased to 53.5 percent for the last or third oxidation stage. Lower Banner coal in 80:20 and 70:30 blends with Pocahontas No. 3 contracted 11.5 and 6.0 percent, respectively. Both of these blends should be safe for industrial use, since neither expanded under a constant applied load of 2.2 pounds per square inch during carbonization in the sole-heated oven.

Two additional samples of Lower Banner coal (c91 and d91), which represented other parts of the No. 56 mine, contracted less than the carbonization sample.

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TABLE 19. - Comparison of results from three types of expansion ovens

Coal XP-38 - 85 percent Pittsburgh bed and 15 percent Upper Kittanning bed
Coal 235 - From coal-charging car at coke oven, Rosita, Coahuila, Mexico
Coal XP-56A - 80 percent Pittsburgh bed and 20 percent Pocahontas No. 3 bed
Coal 82D - 35 percent Eagle bed, 35 percent Powellton bed, and 30 percent Pocahontas No. 3 bed
Coal 87A - 80 percent Pittsburgh bed and 20 percent Hill bed (washed)
Coal 89 - 100 percent Pocahontas No. 6 bed
Coal 257 - 49.5 percent Eagle bed and 50.5 percent Pocahontas No. 3 bed
Coal 81A - 80 percent Powellton bed and 20 percent Pocahontas No. 3 bed
Coal XP36 - 100 percent Mary Lee bed

	Sole-heated oven	Large vertical oven			Small vertical oven		
Coal No.	Average maximum expansion at bulk density of 55.5 lb. per cu. ft., percent	Bulk density, lb. per cu. ft.	Maximum pressure, lb. per square inch		Bulk density, lb. per cu. ft.	Maximum pressure, lb. per square inch	
			Wall	Gas		Wall	Gas
XP-38	-	53.1	0.7	0.7	48.4	0.4	0.6
235	-12.7	51.5	1.2	1.3	1/50.1	2.6	3.1
XP 56A	- 6.4	49.8	1.5	1.8	1/49.6	1.9	4.2
82D	- 3.6	49.0	1.5	2.4	1/50.7	3.2	6.5
87A	- 1.5	50.7	1.7	-	1/50.3	2.9	5.0
89	+ .2	-	-	-	1/50.1	4.3	8.5
257	+ 2.1	1/49.9	3.6	4.4	1/2/50.0	4.6	9.5
81A	+ 3.2	50.7	2.2	3.4	49.2	5.6	12.0
XP-36	+ 4.4	3/51.8	2.2	3.3	4/49.3	4.3	9.0

1/ Tested at 2,000°-2,200° F. heating schedule; all other tests in both large and small vertical ovens made at 2,300°-2,500° F. heating schedule.
2/ Single test only.
3/ Moisture content, 8.7 percent.
4/ Moisture content, 1.5 percent.

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TABLE 20. - Description of coals and blends tested[1/]

Coal No.	Description
m28	Pittsburgh bed, Warden mine (washed)
d75	Pocahontas No. 3 bed, Carswell mine, Kimball, McDowell County, W. Va.
91	Lower Banner bed, No. 56 mine, Dante, Russell County, Va. Breakthrough between 3d and 4th mains, N. 1,420 feet, W. 290 feet from drift mouth.
c91	Special expansion sample of coal 91 from first right, 400 feet off fourth main.
d91	Special expansion sample of coal 91 from No. 6 room, 239 feet off first left moist coal face.
91A	80 percent coal 91 and 20 percent coal d75.
91B	70 percent coal 91 and 30 percent coal d75.
291A	50 percent coal 291 and 50 percent coal c75.
291B	70 percent coal 291 and 30 percent coal c75.
296	Sayreton run-of-mine on which float-and-sink has been made and determination for ash analyzing 10.30.
297	Sayreton No. 2 washed and crushed to approximately 68 percent minus 1/8-inch mesh.
298	Sayreton fine coal. Sampled and screened with 60 percent through 1/8-inch mesh. Sample then through hammer mill and screened, with approximately 75 percent through 1/8-inch mesh.
299	Sayreton coarse coal. Sampled and screened with 30 percent through 1/8-inch mesh. Sample then through hammer mill and screened, with approximately 75 percent through 1/8-inch mesh.
300	Sayreton average coal. Sampled and screened, with 45 percent through 1/8-inch mesh. Sample then through hammer mill and screened, with approximately 75 percent through 1/8-inch mesh.
317	Pocahontas No. 3 bed.
317A	20 percent coal 317 and 80 percent coal m28.
304	Morgantown, W. Va., Ordnance sample 1. Mixture of coal from Rosedale mine and McCartney mines, Monongalia County, W. Va.
305	Morgantown, W. Va., Ordnance sample 2. Composed of same coals as 304.
306	Morgantown, W. Va., Ordnance sample 3. Composed of same coals as 304.
310A[1/]	Blend of Argentine asfaltitas. 50 percent 310 and 50 percent a310.

1/ See table 11 for description of coals 291, 292, 293, 294, 295, 308, 309, 310, 310A, 312, and 313.

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TABLE 21. - Expansion test results in sole-heated oven

Coal No.	Moisture, percent	Expansion, percent		
		Calculated to 55.5 lb. per cu. ft.		
		As tested	Dry basis	Dry solid coal
m28	1.6	-14.0	-12.6	30.2
m281/	1.9	- 7.7	- 5.9	39.6
m282/	1.9	- 5.9	- 4.1	42.8
m283/	-	-10.2	-	-
175	2.8	+19.7	+23.1	89.0
91	1.5	-25.8	-24.7	11.5
914/	1.7	-17.8	-16.4	24.1
915/	1.4	-16.9	-15.7	25.8
916/	1.7	-16.1	-14.6	27.8
c91	1.5	-25.5	-24.4	12.3
d91	2.5	-22.2	-20.2	17.9
91A	1.6	-11.5	-10.2	34.8
91B	1.6	- 6.0	- 4.5	43.7
291	2.4	-12.7	-10.5	35.3
291A	2.0	+ 7.1	+ 9.2	65.8
291B	2.2	+ 1.5	+ 3.7	56.4
292	2.7	-14.6	-12.2	33.7
293	3.3	-16.6	-13.8	29.3
294	3.5	-15.3	-12.2	32.1
295	4.0	-11.1	- 7.4	41.4
296	1.3	- 7.6	- 6.4	44.2
297	2.2	- 2.2	.0	53.2
298	5.9	- 8.9	- 3.2	48.6
2987/	.9	- 4.2	8/- 3.4	48.4
299	5.9	- 7.4	- 1.6	50.3
2997/	.9	- 2.6	8/- 1.7	50.0
300	6.2	- 8.3	- 2.3	49.8
3007/	1.1	- 2.6	8/- 1.7	50.8
308	4.6	- 2.9	+ 1.8	55.1
309	7.2	- 7.3	- .1	52.4
312	6.8	- 4.3	+ 2.6	58.0
313	6.7	-11.3	- 4.9	47.0
317	4.3	+ 4.9	+ 9.6	-
317A	1.9	- 5.6	- 3.8	44.1
304	4.0	-26.1	-23.0	18.5
305	4.5	-29.9	-26.6	15.1
306	3.9	-30.7	-27.8	12.0

1/ Oxidized 3.2 days in air at 100° C.
2/ Oxidized 3.0 days in air at 100° C.
3/ Oxidized 6.0 days in air at 100° C.
4/ Oxidized 3.9 days in air at 100° C.
5/ Oxidized 11.2 days in air at 100° C.
6/ Oxidized 19.2 days in air at 100° C.
7/ Coal dried in laboratory.
8/ Not maximum expansion. Test bulk densities were greater than 55.5 lb. per cu. ft., which gave positive expansions during part of the test.

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Expansion tests were made on coals 291 to 300, inclusive, 308, 309, 312, and 313, and all contracted at 55.5 pounds per cubic foot. Sample Nos. 298, 299, and 300 were tested also after being dried. Comparison of the results obtained on air-dried and as-received coal indicate that the lower-moisture coal expanded more. If the expansion of both samples at 55.5 pounds per cubic foot are calculated on the weight of dry coal per cubic foot, the expansion of both dried and undried samples is about the same. The differences are of the order of the deviation between duplicate tests.

Pocahontas No. 3-bed coal (317) expanded 4.9 percent at 55.5 pounds per cubic foot, which is low for that coal. Other expansion results previously determined in the sole-heated oven for Pocahontas No. 3 bed coals are: Buckeye No. 3 mine, Wyoming County, W. Va. (No. 56), 17.8 percent, and Carswell mine, McDowell County, W. Va. (No. 75), 24.4 percent. The 80:20 blend of Pocahontas No. 3 (No. 317) and Pittsburgh (m28) coals contracted 5.6 percent.

Plasticity of Coals

Plastic properties of 30 fresh coals, 15 oxidized coals, and 5 coal blends described in table 22 were determined during the fiscal year. Excepting coals 314, 315, and 316, all samples were tested by the Gieseler plastometer method. The nine coals oxidized at 99.3° C., the eight Canadian coals, and the four South American coals were not tested by the Davis plastometer method. Two or more tests on the same sample were usually made by the method used. A total of 193 tests - 117 by the Gieseler method and 76 by the Davis method - were made.

TABLE 22. - Description of coals and blends tested

Coal No.	Description
88A	80 percent No. 5 Block Bed, No. 5 mine, Raleigh County, W. Va., and 20 percent Pocahontas No. 3 bed, McDowell County, W. Va. (coal c75).
88B	70 percent No. 5 Block bed, No. 5 mine, Raleigh County, W. Va., and 30 percent Pocahontas No. 3 bed, McDowell County, W. Va. (coal c75).
91	Lower Banner bed, No. 56 mine, Russell County, Va.
c91	Lower Banner bed, first right, 400 feet off 4th main.
d91	Lower Banner bed, No. 6 room, 239 feet off 1st left, moist coal face.
d75	Pocahontas No. 3 bed, McDowell County, W. Va.
91A	80 percent coal 91 and 20 percent coal d75.
91B	70 percent coal 91 and 30 percent coal d75.
92	Upper Banner bed, No. 9 mine, Dickenson County, Va.
92A	3.2 percent Lower Banner bed (coal b91, resample 91), 36.8 percent Lower Banner bed (coal a91, resample 91), and 60 percent coal 92.
m28	Pittsburgh bed, Warden mine, Allegheny County, Pa. (washed).
291	Thick Freeport bed, Indianola mine, Allegheny County, Pa.
292	Thick Freeport bed, Russellton mine, Allegheny County, Pa.
293	Pittsburgh bed, Crescent mine, Washington County, Pa.

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TABLE 22. - Description of coals and blends tested (Cont'd.)

Coal No.	Description
294	Pittsburgh bed, Clyde mine, Washington County, Pa.
295	Elkhorn bed, Republic Kentucky mine, Pike County, Ky.
308	Mary Lee bed, Sayreton No. 2 mine, Jefferson County, Ala.
309	Mary Lee bed, Sayreton Nos. 1 and 2 mines, Jefferson County, Ala.
312	America bed, Virginia mine, Jefferson County, Ala.
313	Mary Lee bed, Sayre mine, Jefferson County, Ala.
314	"B" or Lower Kittanning bed, Pennsylvania No. 46 mine, Winburne, Pa.
315	Fairview bed, Sutt Creek mine, St. Clair County, Ala.
316	Corona bed, Corona No. 20 mine, Warrior Field, Ala.
	Oxidized Samples
	Stored on open trays at room temperature for days indicated:
h28-5a-ox	Pittsburgh bed, Warden mine, Allegheny County, Pa. (washed), 1,303 days.
78-6a-ox	McAlester bed, Bernardi mine, Pittsburg County, Okla., 1,291 days.
81-4a-ox	Powellton bed, Coal Mountain mine, Wyoming County, W. Va., 1,136 days.
82-4a-ox	Eagle bed, Carbon Fuel Co. mine, Kanawha County, W. Va., 1,211 days.
86-2a-ox	Elkhorn No. 3 bed, Wheelwright mine, Floyd County, Ky., 788 days.
156-7a-ox	Raton bed, Brilliant mine, Colfax County, N. Mex., 1,364 days.
	Oxidized at 99.3° C. for days indicated:
m28-1-ox	Pittsburgh bed, Warden mine, Allegheny County, Pa., 3.2 days, small unit.
m28-1-ox	Pittsburgh bed, Warden mine, Allegheny County, Pa., 3.0 days.
m28-2-ox	Pittsburgh bed, Warden mine, Allegheny County, Pa., 6.0 days.
91-1-ox	Lower Banner bed, No. 56 mine, Russell County, Va., 3.9 days.
91-2-ox	Lower Banner bed, No. 56 mine, Russell County, Va., 11.2 days.
91-3-ox	Lower Banner bed, No. 56 mine, Russell County, Va., 19.2 days.
92-1-ox	Upper Banner bed, No. 9 mine, Dickenson County, Va., 3.3 days.
311-1-ox	No. 9 bed, Santa Maria mine, Naricual Coal Basin, Venezuela, 3.2 days.
b310-1-ox	Sample 3, asfaltita from Argentina, S. A., 3.3 days (not a true coal)
	Canadian Coals
28081	Phalen bed, No. 1B mine, Nova Scotia.
28082	Phalen bed, No. 4 mine, Nova Scotia.
28083	Phalen bed, No. 16 mine, Nova Scotia.
28084	Harbour bed, No. 12 mine, Nova Scotia.
28085	Harbour bed, No. 26 mine, Nova Scotia.
28086	Harbour bed, Princess mine, Nova Scotia.
28087	Emery bed, No. 11 mine, Nova Scotia.
28088	Gardiner bed, No. 25 mine, Nova Scotia.
	South American Coals
310	Run-of-mine asfaltita from Argentina (not a true coal).
a310	Sample No. 2, asfaltita from Argentina (not a true coal).
b310	Sample No. 3, asfaltita from Argentina (not a true coal).
311	No. 9 bed, Santa Maria mine, Naricual coal basin, Venezuela.

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The 80:20 and 70:30 blends of high-volatile A coal 88 and low-volatile coal d75 showed plastic properties typical of blends of these compositions. Raising the proportion of the low-volatile coal from 20 percent to 30 increased the characteristic temperatures observed by the two test methods, decreased the maximum fluidity (Gieseler method), and increased the maximum resistance (Davis method). The three samples from Lower Banner bed, coals 91, c91, and d91, taken from different points in the mine, showed differences in plastic properties. Coal c91 was most fluid and coal 91 least fluid; it also showed the highest maximum resistance. Characteristic temperatures agreed well for the three coals, except for the end of resistance, for coal 91 which was appreciably higher. Pocahontas No. 3-bed coal (d75) showed plastic properties typical of other samples of this low-volatile coal tested earlier; a high maximum resistance of 83.4 pound-inches was developed in the Davis plastometer test. Blends of this coal with 80 and 70 percents of high-volatile coal 91 gave plastic properties typical of the proportions used, the 80:20 blend being most fluid. Coal 92 is high-volatile A bituminous in rank; its dry, mineral-matter-free fixed carbon content is 67.4 percent compared with 62.1 percent for coal 91. It swelled strongly in the Gieseler plastometer test, and its maximum fluidity was only half that of coal 91. The blend containing both coals (sample 92A) gave plastic properties which indicate that blending improves coking properties. Coal m28 is a resample of the Warden mine and is used as a standard high-volatile A blending coal in the BM-AGA Survey of American Coals. Its plastic properties are typical of a coal of this rank and agree well with other samples from this mine tested earlier. The plastic properties of the two high-volatile A Thick Freeport-bed coals 291 and 292 are much alike; both coals showed a maximum fluidity of 12,000 dial divisions per minute, strong swelling in the Gieseler test, and low maximum resistance in the Davis test. In comparison, the two Pittsburgh-bed coals 293 and 294 of about the same dry, mineral-matter-free fixed-carbon content were less fluid; coal 294 was more fluid than coal 293. Elkhorn-bed coal 295 is a higher-ranking high-volatile A coal than the Thick Freeport and Pittsburgh-bed coals. The higher rank is reflected in its plastic properties; its maximum fluidity is lower, and its characteristic temperatures and maximum resistance values are higher than of the lower-ranking coals. The three Mary Lee-bed coals - 308, 309, and 313 - are high-ranking high-volatile A coals and showed plastic properties characteristic of this rank. All showed strong swelling in the Gieseler test; coal 313 was the most fluid. The American-bed coal is medium-volatile in rank and showed only moderate swelling, lower fluidity, and higher characteristic temperatures and maximum resistance than the other Alabama coals. These differences would be expected from the relative ranks of the coals from the two different beds. Coals 314, 315, and 316 are high-volatile A bituminous, decreasing in rank in the order named. They were tested only in the Davis plastometer and showed decreasing characteristic temperatures and decreasing maximum resistance in this same order.

The six high-volatile A coals that were stored on open trays at room temperature for the number of days indicated before testing their plastic properties are difficult to compare directly because the rate of loss of carbonizing properties with oxidation differs for individual coals. At the times indicated, coal 82-4a-ox showed strong swelling and the highest maximum

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fluidity in the Gieseler test and the highest maximum resistance in the Davis test of the six coals. Coal h28-5a-ox showed moderate swelling and the other four coals little or no swelling in the Gieseler tests. Considering the days of storage and data obtained in another investigation, the plastic properties obtained on the six coals were characteristic of those to be expected.

The nine coal samples that were oxidized at 99.3° C. for the number of days indicated before testing in the Gieseler plastometer all showed that oxidation had affected their plastic properties. Reduction in swelling and increase in maximum fluidity, with mild oxidation of the Warden mine coal, suggest that its carbonizing properties would be improved; this prediction is verified from data obtained on other samples of this coal. The maximum fluidity of Lower Banner, Upper Banner, and No. 9-bed coals was reduced by oxidation, whereas that of the asfaltita sample (not a true coal) increased. The unusually large increase, from 3,670 to 17,500 dial divisions per minute, after oxidation for 3.3 days may be partly explained by the fact that the coke residue left after the test had a very unusual cell structure and oxidation has caused the asphaltic material to undergo a marked change.

The eight Canadian coals from Nova Scotia were received for test from the Canadian Bureau of Mines. These high-volatile A bituminous coals all showed strong swelling in the Gieseler plastometer tests. The maximum fluidity values ranged from a low of 1,450 dial divisions per minute for the Gardiner-bed coal to a high of 10,000 dial divisions per minute for the Harbour-bed, No. 12 mine, coal. Characteristic temperatures and fluidity values for all eight coals indicate that, so far as plastic properties are concerned, all coals would make good coke.

Tests of the plastic properties of the three asfaltita samples showed that samples 2 and 3 were much more fluid than the run-of-mine sample. Samples 2 and 3 have dry, mineral-matter-free fixed carbon contents of 54.2 and 53.6 percent, respectively, compared with 68.4 percent for the run-of-mine sample. The latter fused very weakly, giving a fluidity of only 1.1 dial divisions per minute compared with 5,000 and 3,670 dial divisions per minute for samples 2 and 3, respectively. Coal 311 from Venezuela contained 54.8 percent of dry, mineral-matter-free fixed carbon and had a heating value of 14,060 B.t.u. per pound on the moist, mineral-matter-free basis. This low-ranking high-volatile A bituminous coal showed a maximum fluidity of only 2.2 dial divisions per minute, which is characteristic of many coals of this low rank when they have a high oxygen content.

Oxidizing Properties of Various Bituminous Coals

The characteristic rate of oxidation of coal is defined as the percentage of oxygen by weight of the coal after the coal has consumed 1 percent of oxygen by weight; the numerical value may be expressed either on the basis of dry, mineral-matter-free coal or on the basis of dry coal. To determine this rate, the coal is subjected to oxidation at constant temperature (99.3° C.) in a specially designed apparatus and under defined test conditions that give data from which the relation between amount of oxygen consumed and time of

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oxidation may be calculated. Two different sizes of apparatus, specially designed and built for controlling the oxidation of the coal at 99.3° C., are employed: (1) A 450-pound-capacity, steam-jacketed, rotary-drum unit and (2) a similar unit of 50-pound capacity. Both units use a controlled supply of air after the coal charge has been dried in situ using nitrogen.

Table 23 summarizes the results obtained on 13 coals tested during the year. The coals are listed in the approximate order of decreasing rank within each of the two size ranges of coal tested. Coal 296 contained 11.3 percent ash on the dry basis, which accounts for its extremely low characteristic rate of oxidation, as compared with rates for the other 4 Alabama coals. One might expect that coal 300 would show a characteristic rate of oxidation between those for coals 299 and 298. The relative freshness of the 3 samples was unknown; freshness of preparation of sample before testing is an important factor in the rate of oxidation. Coal DSC-2, as received, was a grab sample from the top bench and contained 9.2 percent ash and 2.4 percent sulfur. Its lower rate of oxidation, as compared with rates for coals 292 and 291, which meet the specifications for gas and coking coals and are representative of their respective mines, would be expected. Duplicate tests on coals DSC-2 and 292 were made to determine the reproducibility of tests in the small drum unit; more work needs to be done to establish this reproducibility with certainty.

Because of the growing industrial practice in recent years of using finer size coal in coke-oven charges, 0- to 1/8-inch, stage-crushed charges were used in tests of the last five samples listed in the table. The results obtained were as expected for coals 92, 91, m28, and 311. Close reproducibility of results was obtained on coal m28 by the large and small test units. As the name implies, sample b310 contained asphaltic material and is not a true coal. It contained only 53.6 percent dry, mineral-matter-free fixed carbon but had a heating value of 16,630 B.t.u. per pound on the moist, mineral-matter-free basis. Its unusual composition in respect to material contributing to a high treating value accounts for its relatively low rate of oxidation, as compared with the other four coals tested in the same size range.

The last 2 columns of the table show the relative rates of oxidation of the 13 coals compared to the Pittsburgh bed, Warden mine, coal taken as unity. The first 8 coals are compared for the 0- to 1/4-inch, stage-crushed size range; the last 5 samples for the 0- to 1/8-inch, stage-crushed size range.

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TABLE 23. - Oxidizing properties of various bituminous coals

Coal 296 - Mary Lee bed, Sayreton No. 1 mine, Jefferson County, Ala., run-of-mine coal on which float-and-sink was made, high-ash, crushed to approximately 68 percent through 1/8-inch.
Coal 297 - Mary Lee bed, Sayreton No. 2 mine, Jefferson County, Ala., washed coal crushed to approximately 68 percent through 1/8-inch.
Coal 299 - Mary Lee bed, Sayreton coarse coal, 30 percent through 1/8-inch, crushed to approximately 75 percent through 1/8-inch.
Coal 298 - Mary Lee bed, Sayreton fine coal, 60 percent through 1/8-inch, crushed to approximately 75 percent through 1/8-inch.
Coal 300 - Mary Lee bed, Sayreton average coal, 45 percent through 1/8-inch, crushed to approximately 75 percent through 1/8-inch.
Coal DSC-2 - Pittsburgh bed, Shannopin mine, Green County, Pa.
Coal 292 - Thick Freeport bed, Russellton mine, Allegheny County, Pa.
Coal 291 - Thick Freeport bed, Indianola mine, Allegheny County, Pa.
Coal 92 - Upper Banner bed, No. 9 mine, 0- to 1-1/4-inch raw nut and slack from tipple, Dickenson County, Va.
Coal 91 - Lower Banner bed, No. 56 mine, Russell County, Va.
Coal m28 - Pittsburgh bed, Warden mine, Allegheny County, Pa., washed coal.
Coal b310 - Asfaltita, sample No. 3, Argentina, S. A. (not a true coal).

Coal No.	Stage-crushed coal, size range tested	Drum unit used	Characteristic oxidation rate; absolute values		Oxidation rate relative to Warden mine coal 1/	
			Dry, m.m.-free coal	Dry coal	Dry, m.m.-free	Dry
296	0- to 1/4-inch	Small	0.062	0.054	0.47	0.43
297	do.	do.	.101	.089	.76	.70
299	do.	do.	.109	.097	.82	.76
298	do.	do.	.126	.110	.95	.75
300	do.	do.	.167	.148	1.26	1.17
DSC-2	do.	do.	.140	.126	1.05	.99
Do.	do.	do.	.219	.196	1.65	1.54
292	do.	do.	.209	.188	1.57	1.48
Do.	do.	do.	.275	.247	2.07	1.63
291	do.	do.	.301	.275	2.26	1.38
92	0- to 1/8-inch	do.	.121	.110	.69	.67
91	do.	Large	.157	.146	.89	.88
m28	do.	do.	.176	.165	1.00	1.00
Do.	do.	Small	.186	.174	1.05	1.05
311	do.	do.	.300	.293	1.70	1.78
b310	do.	do.	.149	.143	.85	.87

1/ Absolute values for 0- to 1/4-inch size Pittsburgh-bed, Warden mine coal are 0.133 on the dry, mineral-matter-free basis and 0.127 on the dry basis; corresponding values for 0- to 1/8-inch size are 0.176 and 0.165.

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Low-Temperature Coke by Krupp-Lurgi Process

Although only one low-temperature carbonization plant has been operated commercially in the United States on bituminous coal, the problem of low-temperature carbonization and the utilization of the products continue to intrigue research workers and coal-producing companies. As a member of the Solid Fuels Mission to Germany, one of the Bureau of Mines' staff visited two German commercial-size plants of Krupp-Lurgi design and published his observations.50/ Carbonization is conducted in rectangular cells 10 feet 6 inches long, and 6 feet 11 inches high, with an average width of 3-1/2 inches. To facilitate removal of the product, the cells are 3 inches wide at the top and 4 inches at the bottom. Gases are recirculated to heat the charge. These gases enter the oven at about 620° C. and have a velocity so that the exit gases are not more than 50° cooler than the entering gases. The time for a complete cycle in the Krupp-Lurgi plant averages between 5 hours 20 minutes and 6 hours. The ovens were equipped for byproduct recovery. The low-temperature coke was used in locomotives as a gas generator fuel in stationary Diesel plants, for the production of synthesis gas for synthetic liquid fuels. The 3/4-inch to 1-1/2-inch size made an excellent fuel for ordinary household stoves. The author concluded his report with:

> It should be kept in mind, when considering the economic practicability of the low-temperature carbonization process, that it produces a better domestic fuel than high-volatile bituminous coal, and that this better fuel costs more per B.t.u. than the raw material from which it was made. This increase in cost per B.t.u. depends on the size of the plant, the local conditions, and the quality of the coal. All these factors can vary through a considerable range.

Carbonization of Elkhorn No. 3-Bed Coal

An investigation of Elkhorn No. 3-bed51/ coal from Wheelwright mine, Floyd County, Ky., showed this coal to be of high-volatile A rank, low in ash and sulfur, and suitable for gas making. Its coke was fissured excessively, but stronger coke was obtained from blends containing 20 and 30 percent Pocahontas No. 3 coal. The carbonization sample contracted 7.4 percent at a charge density of 51.92 pounds per cubic foot; expansion tests indicated that blends containing as much as 30 percent Pocahontas No. 3 coal may be carbonized without damage to byproduct ovens. The characteristic oxidation rate of Elkhorn No. 3 coal was about twice that of Pittsburgh-bed coal.

50/ Schmidt, L. D., Low-Temperature Coke by the Krupp-Lurgi Process: Bureau of Mines Inf. Circ. 7395, 1947, 18 pp.

51/ See footnote 47, page 61.

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Carbonizing Properties of Western Coals

In an investigation of the properties of western coals,52/ 106 coals from 7 Western States were subjected to carbonization or related tests. These coals were classified by States as follows: California, 2; Colorado, 24; New Mexico, 12; Texas, 2; Utah, 39; Washington, 5; and Wyoming, 23. They ranged in rank from subbituminous C through high-volatile A bituminous, although a major proportion were bituminous coals. The high-volatile A coals generally contained more volatile matter and oxygen than eastern coals of similar rank; consequently, their coking properties were inferior. The correlation of agglutinating value with coking propensity was more satisfactory for these western coals than for Appalachian coals. Many samples did not fuse in Gieseler and Davis plastometer tests at the normal heating rate of 3° C. per minute. Generally, the yields of gas were high and those of coke low.

Two California coals that outcrop in the Eel River district were of subbituminous rank and gave low yields of noncoherent char and high yields of gas at 950° C. The composition of Colorado coals varied greatly. Twenty-two ranked as high-volatile bituminous, but 12 of these were low in that classification and coked weakly. Frederick coal from the Trinidad district was the only Colorado coal fusing in plastometer tests, and it had the highest agglutinating value (8.5 kilograms) of any western coal tested. It yielded well-fused coke that was stronger than the average coke from eastern high-volatile A coals. Oliver and Hawks Nest mine coals from Gunnison County coke less strongly than Lower Sunnyside coal from the Columbia mine in Carbon County. Thirteen core-drill samples from the Paonia district, Gunnison County, yielded either noncoherent char or friable coke.53/ Eight New Mexico coals were of high-volatile A rank, and four were high-volatile C coals. Three Raton-bed coals from Colfax County that had agglutinating and plastic properties characteristic of high-volatile coking coals, yielded abradable coke. Two Gallup district coals were noncoking. Miller Gulch- and Lamb-mine coals from Santa Fe County coked as strongly as Lower Sunnyside (Utah) coal, and Jones mine coal from the same county coked even more strongly. One sample from Socorro County coked weakly. Two high-volatile coals from Texas were almost nonagglutinating and noncoking. Thirty-six, or all but 2, Utah coals were of bituminous rank. Upper and Lower Sunnyside-bed coals from the Horse Canyon and Sunnyside mines yielded coke similar to that from Columbia mine coal, the coking properties of which are well-known. Columbia mine coal was carbonized at bulk densities of 50.9, 52.6, 56.1, and 59.3 pounds per cubic foot, neither the yields of products nor the coke strength being affected significantly by the variation in density. Blending Sunnyside mine coal with 10 percent low-temperature char strengthened the coke slightly. Bear Canyon and Blind Canyon coals from Emery County coked weakly and yielded large volumes of gas. Six core-drill samples from Sanpete County coked less strongly than Lower Sunnyside coal. No. 6- and Roslyn No. 5-bed coals from Kittitas County, Wash., were strongly agglutinating and yielded well-fused coke that were rather abradable because they contained appreciable proportions

52/ See footnote 47, page 61.
53/ See footnote 14, page 28.

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of ash. The 23 samples from Wyoming included bituminous and subbituminous coals, most of which were noncoking. BM-AGA tests were made only on Lincoln County coals; 2 samples represented the Gomer mine and 5 were core-drilled. The cokes from the drilled samples had satisfactory shatter indexes but were more abradable than coke from Lower Sunnyside (Utah) coal.

Carbonization of Velva Lignite and Monarch Coal

Velva lignite from Ward County, N. Dak., and Monarch coal from Sheridan County, Wyo.,54/ were carbonized by the BM-AGA method at 500°, 600°, 700°, 800°, and 900° C. Generally, the yields of char, tar, and light oil were low, and the yields of gas and liquor were high. Predrying caused the lignite to devolatilize more completely when carbonized at temperatures above 500° C. Both samples yielded noncoherent char, with size degradation occurring largely below 500° C. The gases had the high specific gravity and low heating values characteristic of gas from low-rank coals.

Improvement of Methods for Testing Physical Properties of Coke

Existing methods for testing coke are inadequate. Coke producers have great difficulty in selecting the better of two coals for coking purposes if they are similar, and coke consumers are in a similar position because methods for closely measuring the physical properties of coke are not in existence.

Investigations are underway at the Southern Experiment Station, Tuscaloosa, Ala., to determine the size of coke sample necessary to obtain physical test data of any desired or specified precision and to improve the precision and utility of tests measuring the resistance of coke to breakage by shattering or impact forces. A mechanized shatter machine, the impact drum, has been developed and preliminary testing is almost complete. Preliminary studies indicate that the mechanized shatter will be more precise than the hand-operated apparatus. They also indicate that mechanization of the apparatus will make practical the development of a testing procedure which will allow the prediction of the amount of breakage a coke will suffer under any given set of handling shocks.

Large-Scale Coking Investigations

A model coke oven, having a capacity of 800 pounds of coal per charge, and the necessary auxiliary equipment are in process of installation at the Southern Experiment Station at Tuscaloosa, Ala. The oven chamber is of full width (17 inches), so that it can be used to study the effect of scale factors in smaller carbonizing apparatus. After the model oven has been subjected to correlation tests with full-scale byproducts ovens, it will be used for investigations of the effect of carbonizing conditions on the quality of coke and for studies of the coking properties of coals available to southern coke producers.

54/ Reynolds, D. A., Davis, J. D., Ode, W. H., Wolfson, D. E., and Birge, G. W., Carbonizing Properties of Velva Lignite from Ward County, N. Dak., and Monarch Coal from Sheridan County, Wyo.: Bureau of Mines Tech. Paper 695, 1946, 41 pp.

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Fuel Industry of Japan - Low-Temperature Carbonization

Between March and September 1946 a field survey was made of the commercial low-temperature carbonization processes used in Japan before and during the war. The results of this investigation were presented at the spring meeting of the American Society of Mechanical Engineers, in Tulsa, Okla., March 2-5, 1947, and were recently abstracted.55/

Although water power was an important source of energy in Japan before the war, a considerable part of the 66,000,000 metric tons of coal mined annually was used for generating steam for power and process industries. A large amount also was used for manufacturing metallurgical coke; and although most of the low-volatile bituminous coal required for blending was at one time imported from north China and Manchuria, Japanese engineers ultimately developed two low-temperature carbonization processes for making a semicoke that suited their blending requirements satisfactorily.

Some of the low-temperature carbonization plants were operated primarily as a source of liquid fuel. Although Japan had a few Bergius and Fischer-Tropsch plants in operation, they were not very successful; but the low-temperature carbonization processes were satisfactory from the start and produced seven times as much liquid fuel as was produced by the synthetic liquid-fuel plants that were in operation. The plants that operated most sucessfully were of German design and consisted of both Lurgi and continuous vertical Koppers units.

The chemical utilization of coal in Japan was primarily for the production of ammonia, and the gasification methods used for producing the necessary synthesis gas were developed largely along conventional lines. The principal source of gas was the Otto-design water-gas set, although Winkler and Fiag gas generators also were used to some extent.

Small-scale low-temperature carbonization plants were used to recover coal tar from high-ash waste coals; it was used directly as a lubricant for mine-car wheels and the pulleys of cable-haulage equipment. Needless to say, its lubricating value was very low, which made it useless for highly loaded bearings.

Very little coal was used in Japan as a domestic fuel, coking and heating being done with small braziers in which semicoke or briquets of semicoke and lime were burned.

During the war, when gasoline was not obtainable for civilian use, the Japanese used a number of portable gas producers for their vehicles. Again, semicoke was the main source of fuel, since charcoal was very scarce; and although the ash frequently was as high as 30 percent, necessitating frequent cleaning of the fuel bed, the vehicles gave staisfactory performance.

55/ Reid, W. T., The Fuel Industry of Japan: Combustion, vol. 18, No. 11, April 1947, pp. 41-42.

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Gases from Carbonization Tests

The large number and varied rank of coals carbonized in the BM-AGA Survey of American Coals since 1929 afford abundant data for correlating properties of coal with those of carbonization products. From correlation of the yields and characteristics of gases[56] obtained from 79 coals at 900° C., the following conclusions were drawn:

1. Yields of gas from low-, medium-, and high-volatile A coals vary with rank of coal on a percentage-by-weight but not on a volume basis.

2. The rank of coal indicates the physical and chemical properties of gas from low-, medium-, and high-volatile A coals: (a) Specific gravity increases with decrease in rank, (b) heating value increases with decrease in rank, and (c) chemical composition changes with decrease in rank - carbon dioxide, illuminants, carbon monoxide, methane, and ethane increase while hydrogen decreases.

3. The rank of coal is unrelated to the hydrogen sulfide content of the gas.

High-B.t.u. Gas from Solid Fuels

In a statement prepared for the natural-gas studies of the Federal Power Commission, the manufacture of high-B.t.u. gas from solid fuels was considered in some detail.[57] It was pointed out that exhaustion of petroleums and natural-gas reserves with time and the ever-possible threat of war clearly dictate a national reserach and development policy looking to the realization of suitable processes for the manufacture of high-B.t.u. gases from solid fuels at levels economically attainable. The paper summarizes very briefly cognate British and European develcpments, including the synthesis of methane, the direct hydrogenation of coal or coke to a high-B.t.u. gas, and the direct manufacture of a high-B.t.u. gas in a steam-oxygen pressure producer. The paper concludes with the following recommendations:

> To implement a policy looking to the manufacture of high-B.t.u. gas from solid fuels, the following topics are suggested for research and development:
>
> (1) The conversion of low-cost, caking American coals into noncaking fuels suitable for water-gas production. This conversion must be made by processes showing high capacities and low investment and low operating costs.

56/ Wolfson, D. E., and Reynolds, D. A., Yields and Properties of Gases from BM-AGA Carbonization Tests at 900° C.: Bureau of Mines Tech. Paper 693, 1946, 12 pp.

57/ Huff, W. J., The Manufacture of High-B.t.u. Gas from Solid Fuels: Federal Power Commission, Natural Gas Investigations Docket G580, Hearings 8, pp. 9618-9641, June 19, 1946; pp. 9908-9924, June 21, 1946.

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(2) The development of water-gas processes of the high-capacity, low-velocity type capable of handling light, small fuels.

As subdivisions under this topic are the examination of (a) continuous oxygen producers and (b) continuous producers of the revolving gas type.

(3) The conversion of blue water gas to a high-B.t.u. methane containing gas in (a) the oxygen-pressure producer; (b) the catalytic synthesis of methane; (c) the direct-pressure hydrogenation of solid fuels.

Of these the first, (a), appears to warrant priority.

Gasification of Buckwheat Anthracite

In cooperation with the Anthracite Institute, who financed the tests, which were planned by the Division of Fuel Technology, The Pennsylvania State College, a study was made on the gasification of Buckwheat No. 2 (Rice) and Buckwheat No. 3 (Barley) sizes of anthracite by means of oxygen.58/ The tests were conducted at Trail, British Columbia, in the plant of the Consolidated Mining & Smelting Co. of Canada, Ltd., during September 1947.

Six cars of Pennsylvania anthracite were gasified, and the test procedure followed was substantially the A.S.M.E. Test Code for Gas Producers.

It was found that synthesis gas can be produced from these two sizes of anthracite having the following typical analyses, in percent:

Constituent	Buckwheat No. 2	Buckwheat No. 3
CO_2	16.50	17.20
O_2	.15	.20
H_2	41.00	37.65
CO	40.00	42.40
CH_4	.85	.75
N_2	1.50	1.80

58/ Wright, C. C., and Newman, L. L., The Oxygen Gasification of Anthracite in the Wellman-Galusha Producer: Presented at Joint Conference of the Production and Chemical Committees of the American Gas Association, New York, June 4, 1947. Abridged in Chem. and Met. Eng., June 1947.

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The salient operating figures are given below:

	Buckwheat No. 2	Buckwheat No. 3
Duration of test .....hr.	25	12
Fuel, as fired .....lb.	69,280	15,919
Gas produced .....M cu. ft.	2,987	614
Fuel gasified .....lb./hr./sq. ft.	33	17
Fuel gasified .....lb./hr.	2,771	1,335
Gas per lb. fuel .....cu. ft.	43.1	38.6
Fuel per M. cu. ft. gas .....lb.	23.2	25.8
O_2 per M cu. ft. gas .....cu. ft.	201	220
O_2 per lb. fuel .....do.	8.66	8.73
Steam used per lb. fuel .....lb.	1.40	1.38
Steam used per M cu. ft. gas .....do.	32.5	35.6
Steam decomposed per lb. fuel .....do.	0.73	0.58
Carbon in dry refuse .....percent	2.6	3.0
Gasification efficiency .....do.	82.8	76.8
Gross heating value of gas ..... B.t.u. per cu. ft.	271	266

Considerable information was obtained relative to operating technique, particularly with respect to the importance of maintaining uniform speed of rotation of the grate to maintain uniform fire conditions. As was expected, the depth of the fire zone was very thin when the Buckwheat No. 3 anthracite was used, with the result that the clinker was denser, and difficulty was experienced with the fire reaching the grate even when it operated at the slowest rates of rotation.

Gasification of Subbituminous Coal and Lignite

Research and development work on gasification of lignite and subbituminous coal in externally heated retorts was continued in the small pilot plant at Golden, Colo., and in the large pilot plant at Grand Forks, N. Dak. Improved coal-handling facilities were installed in the large plant. After one test in this plant it was observed that the welded seams in the Pluramelt alloy retort had failed. Examination revealed that the welds contained only 20.9 percent chromium, which was insufficient to withstand the operating temperatures of 1,900° to 2,000° F. The alloy retort was repaired and made ready to operate, but no further tests were completed in the large plant during the year because of limited personnel and funds.

At Golden, Colo., several tests were made in the small plant to determine the optimum width of the annular reaction zone. A summary report of investigations[59/] was published which described the development of the large plant and all experimental data obtained thereon, with results of work done on the small pilot plant during 1945-46.

59/ Parry, V. F., Gernes, D. C., Wagner, E. O., Goodman, J. B., and Koth, A. W., Gasification of Lignite and Subbituminous Coal, Progress Report for 1945-46: Bureau of Mines Rept. of Investigations 4128, 1947.

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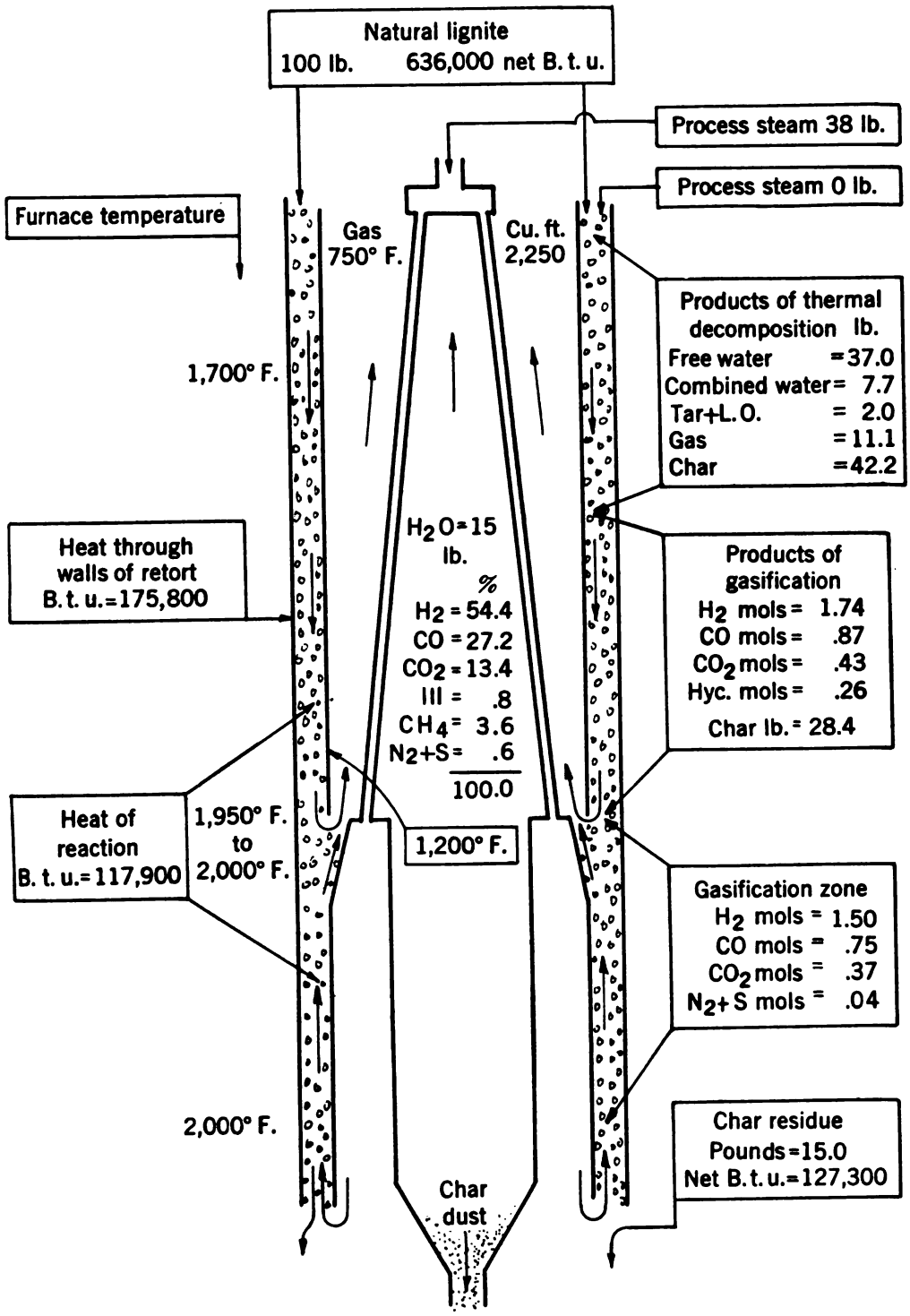

Figure 37. - Mechanism of gasification of natural lignite in externally heated annular retort.

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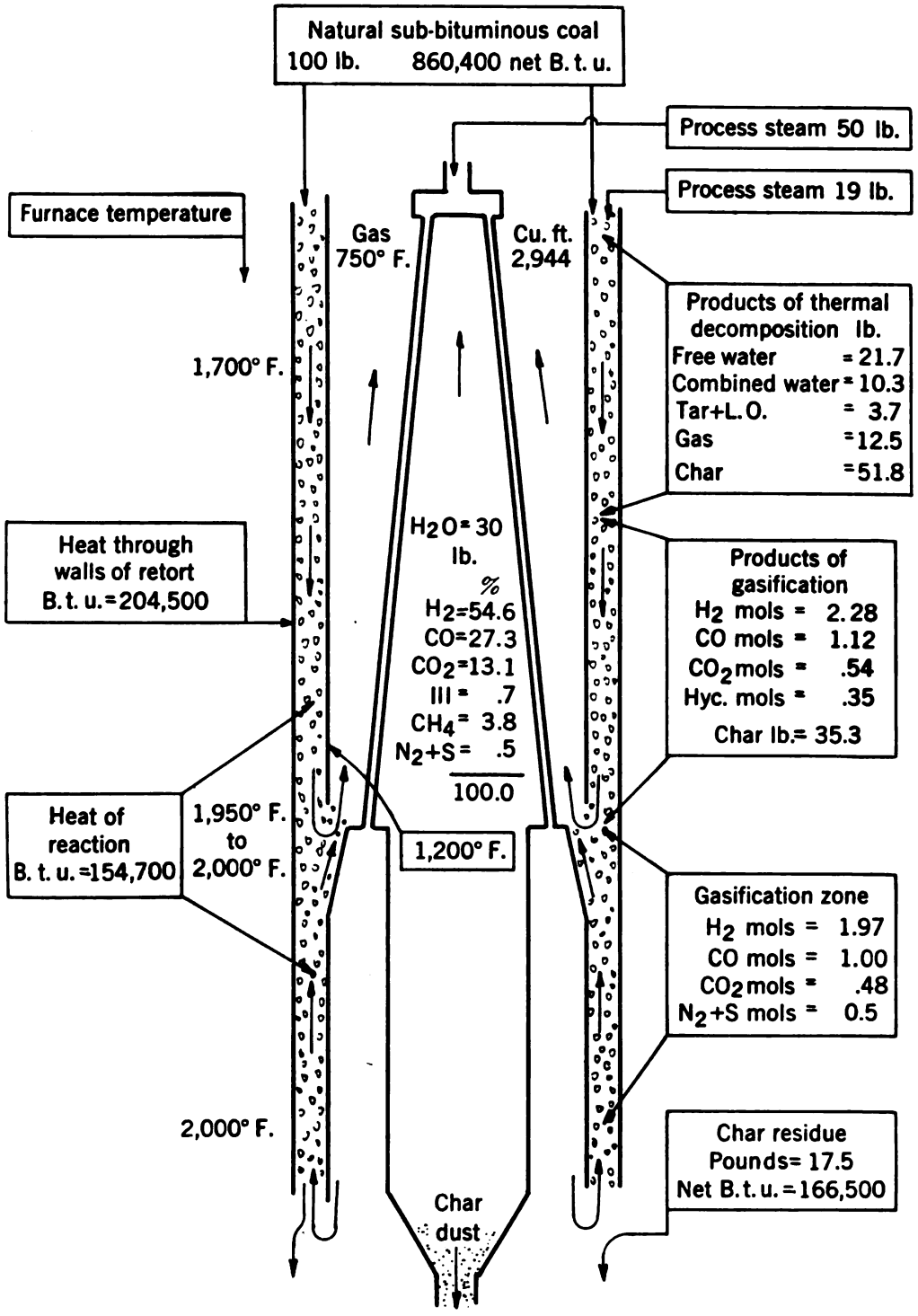

Figure 38. - Mechanism of gasification of natural subbituminous coal in externally heated annular retort.

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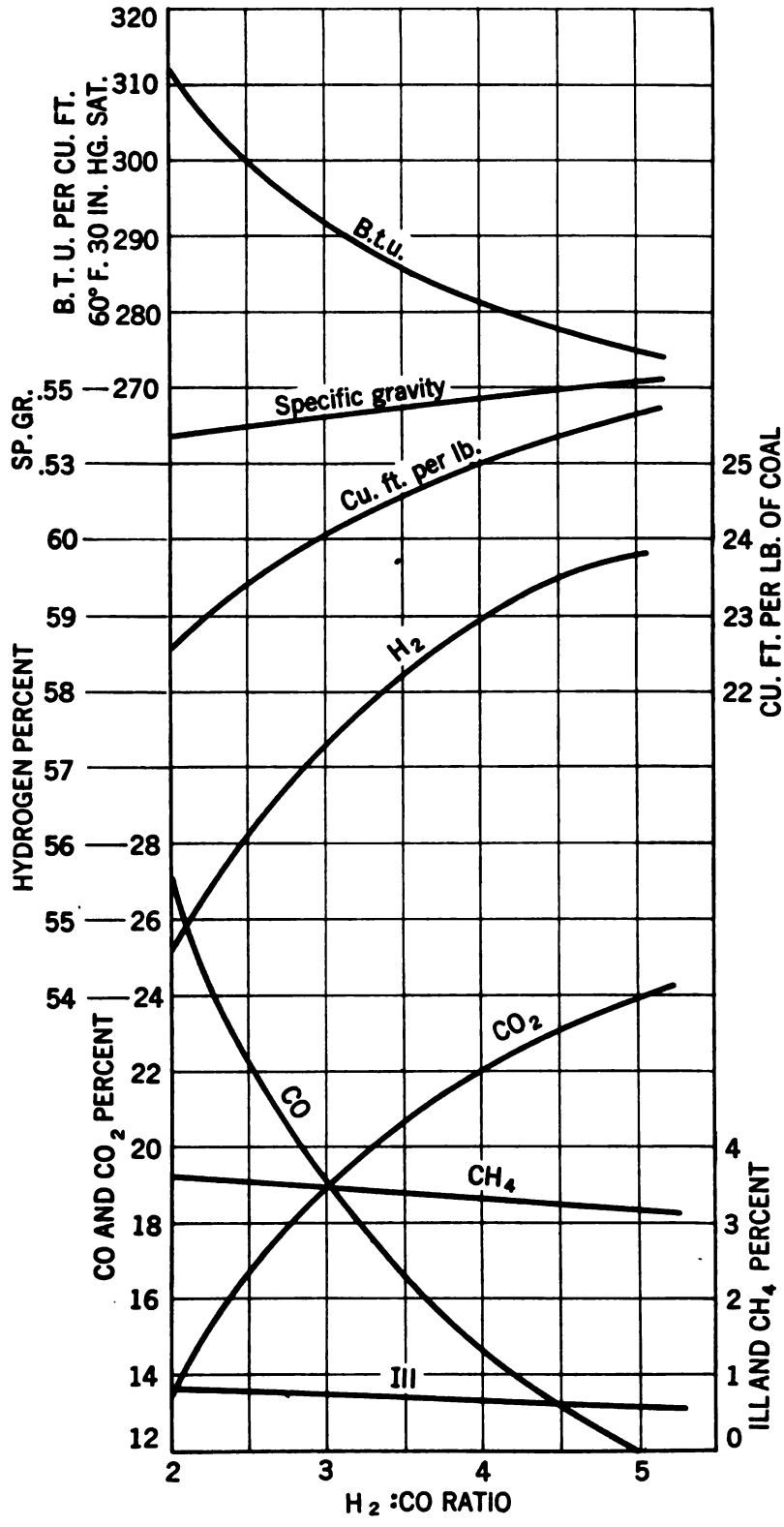

Figure 39. - Gasification of natural lignite (80 percent carbon converted).

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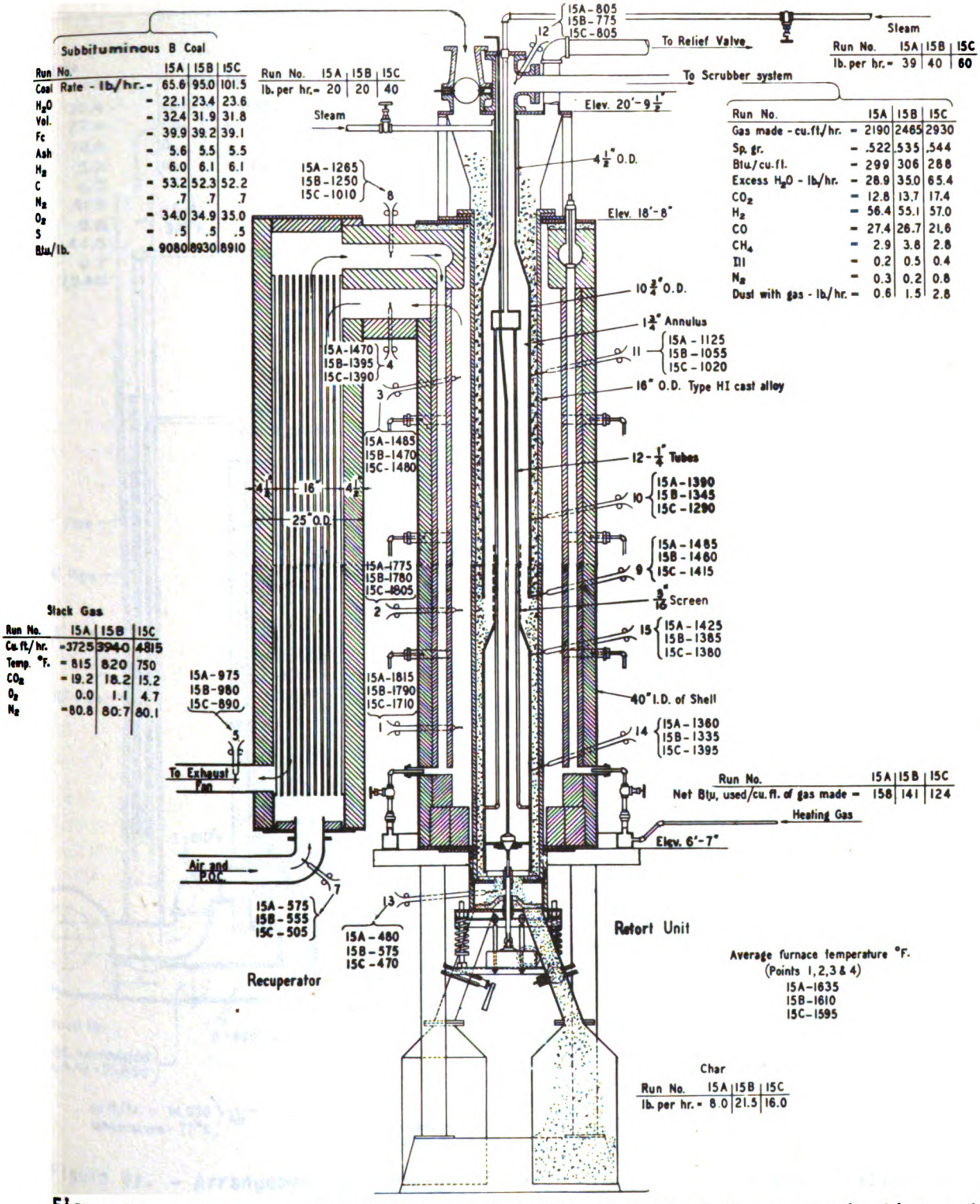

Figure 40. - Arrangement and design of small pilot plant for carbonization and gasification of noncoking coal, Golden, Colo., February 1947.

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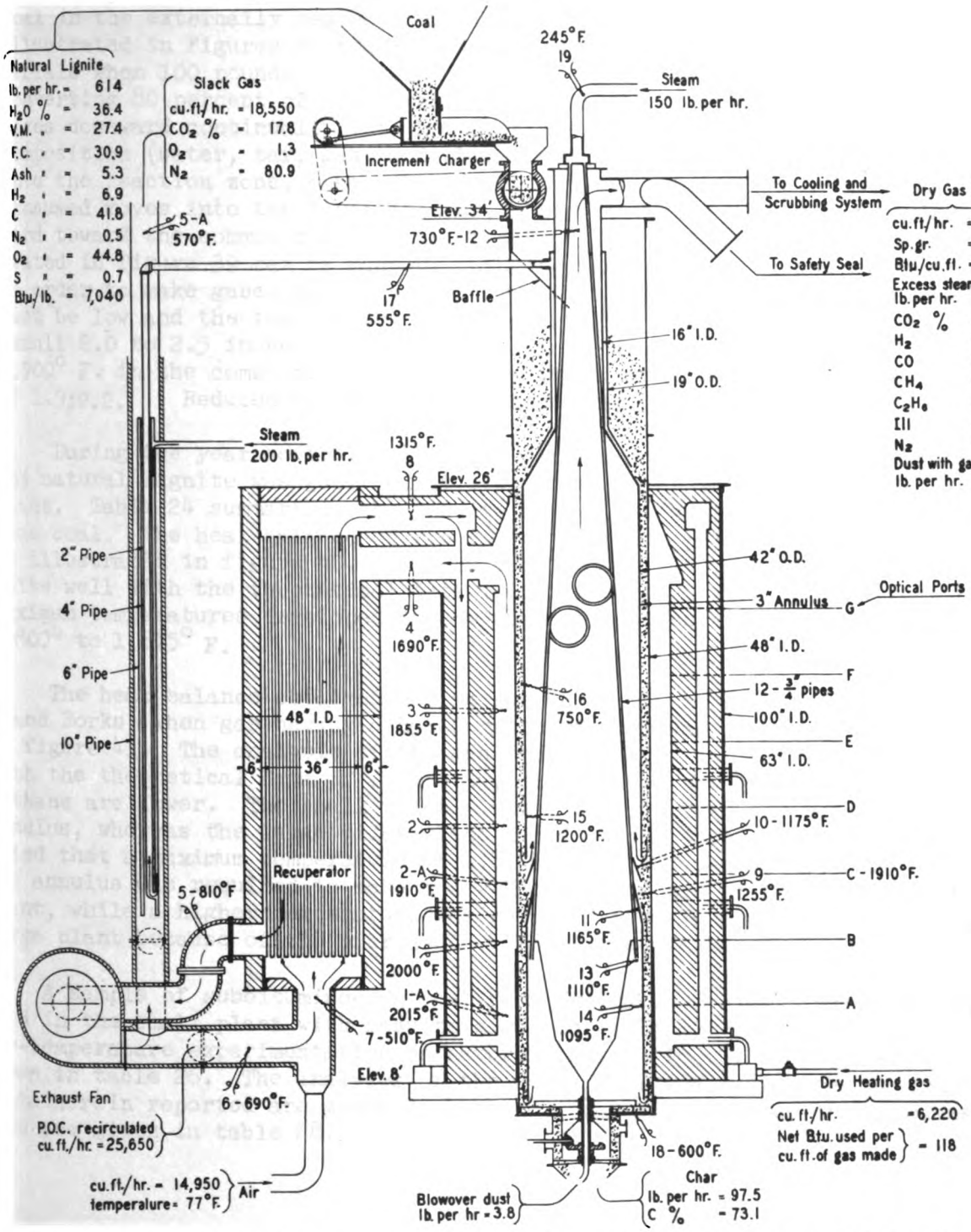

Figure 41. - Arrangement and design of large pilot plant for gasification of lignite, Grand Forks, N. Dak.

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The probable mechanism of gasification of lignite and subbituminous coal in the externally heated annular retort varying amounts of steam is illustrated in figures 37 and 38 which show the theoretical balance of materials when 100 pounds of natural coal is passed through the retort while converting 80 percent of the carbon to gas. Coal entering the upper annulus moves downward continually into hotter zones. The products of thermal decomposition (water, tar, gas, and char) form in the upper section and move into the reaction zone, where they combine to form water gas. The char not consumed moves into the lower annulus, where it reacts with steam moving upward toward the common central gas offtake. The various grades of gas illustrated in figure 39 can be made by changing the temperature and steam flow. In order to make gases having low H_2:CO ratios, the concentration of steam must be low and the temperature relatively high. It has been found that annuli 2.0 to 2.5 inches wide require maximum temperatures of 1,850° to 1,900° F. in the combustion zone to make water gases having H_2:CO ratios of 1.9:2.2. Reduced temperatures produce gases having higher H_2:CO ratios.

During the year several coals were gasified in the small pilot plant, and natural lignite was gasified at low temperatures in the large pilot plant. Table 24 summarizes the results of two tests at Golden on subbituminous coal. The heat balance of run 15 in the small pilot plant at Golden is illustrated in figure 40. It should be noted that runs 15A and 15B check quite well with the theoretical data illustrated in figure 38, and that the maximum temperatures developed in the combustion zone are in the range of 1,800° to 1,815° F.

The heat balance obtained during run 4H on the large pilot plant at Grand Forks, when gas having a H_2:CO ratio of 3.29 was made, is illustrated in figure 41. The analysis of the gas from this run is in fair agreement with the theoretical data of figure 39, except that the illuminants and methane are lower. The small pilot plant (shown in fig. 40) has a 1.75-inch annulus, whereas the large pilot plant has a 3-inch annulus. It should be noted that a maximum temperature of 1,815° F. in the combustion zone outside the annulus was required to make a low-H_2:CO ratio gas in the small pilot plant, while a higher temperature was required to make a similar gas in the large plant because of the increased width of the annulus.

A sample of subbituminous A coal from Hyderabad-Deccan, India, was gasified in the small plant with results given in table 25, and the results of low-temperature experimentation on natural lignite in the large plant are given in table 26. The analyses of the coals gasified during the several tests herein reported are given in table 27, and the analyses of the gases made are given in table 28.

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TABLE 24. - Summary data on gasification of Colorado subbituminous B coal in the Golden, Colo., pilot plant

RUN AND PERIOD NUMBER	(1)	13-A	13-C	15-A	15-C
Date, September 1946	(2)	10-11	15	11-12	13-14
Duration ..... hours	(3)	24	12	24	15
	(4)				
COAL CHARGED ..... lb. per hour	(5)	92	93	65.6	101.5
Moisture as charged ..... percent	(6)	22.8	22.6	22.1	23.6
Ash as charged ..... do.	(7)	5.6	5.6	5.6	5.5
Gasified ..... do.	(8)	82.9	80.4	86.0	71.8
Coal per Mcf of gas ..... do.	(9)	31.6	32.4	30.0	34.6
	(10)				
DRY RESIDUE ..... lb. per hour	(11)	13.6	16.0	8.6	18.8
Char out of bottom	(12)	9.7	13.1	8.0	16.0
Dust with gas	(13)	1.7	1.0	.6	2.8
Ash in total residue ..... percent	(14)	25.4	23.1	46.5	20.7
	(15)				
GAS MADE SGC ..... Mcf per ton	(16)	63.30	61.60	66.77	57.73
McF per hour	(17)	2.91	2.87	2.19	2.93
B.t.u. per cu. ft. observed, gross	(18)	309	308	299	288
B.t.u. per cu. ft. calculated, net	(19)	278	276	267	257
Specific gravity, calculated	(20)	.523	.522	.522	.544
Ratio H_2:CO	(21)	1.97	2.01	2.06	2.64
	(22)				
STEAM USED ..... lb. per hour	(23)				
With coal	(24)	40.6	40.6	20	40
In char zone	(25)	35.2	35.2	39	60
Undecomposed steam ..... lb. per Mcf	(26)	9.6	10.5	13.4	22.7
	(27)				
HEATING SYSTEM DATA	(28)				
Net B.t.u. used per cu. ft. gas made ..	(29)	141	141	158	124
Heat released ..... M B.t.u. per cu.ft.	(30)	22.3	22.2	19.0	19.8
Make gas used ..... Mcf per hour	(31)	1.47	1.47	1.30	1.41
CO_2 in Poc ..... percent	(32)	16.0	16.7	19.2	15.2
Primary air ..... Mcf per hour	(33)	3.72	3.74	3.02	4.01
Poc recirculated .....do.	(34)	5.23	5.54	6.34	7.20
	(35)				
TEMPERATURES ..... °F.	(36)				
Average combustion chamber	(37)	1,745	1,745	1,635	1,595
Bottom of comb. chamber No. 1	(38)	2,000	1,905	1,815	1,710
Middle of comb. chamber No. 2	(39)	1,895	1,905	1,775	1,805
Top of comb. chamber No. 3	(40)	1,565	1,610	1,485	1,480
Outlet from comb. chamber No. 4	(41)	1,515	1,565	1,470	1,390
Inlet to fan 5	(42)	915	970	975	890
Air and Poc to recuperator 7	(43)	495	530	575	505
Air and Poc to furnace 8	(44)	1,200	1,245	1,265	1,010
Gas leaving retort 12	(45)	845	865	805	805
Stack 5a	(46)	775	820	815	750
Gas junction in furnace.....	(47)	1,435	1,450	1,485	1,415
Char zone opposite lower burners	(48)	1,315	1,285	1,360	1,395
	(49)				

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TABLE 25. - Summary data on gasification of subbituminous A coal from Hyderabad-Deccan, India, in Golden, Colo., pilot plant

RUN AND PERIOD NUMBER	(1)	14-A	14-B	14-C	14-D
Date, September 1946	(2)	16	17	17-18	18
Duration ..... hours	(3)	9	12	14	2
	(4)				
COAL CHARGED ..... lb. per hour	(5)	39	105	109	200
Moisture as charged ..... percent	(6)	11.9	12.0	12.4	12.4
Ash as charged ..... do.	(7)	16.3	16.3	16.2	16.2
Gasified ..... do.	(8)	86.1	51.4	52.1	34.6
Coal per Mcf of gas ..... lb.	(9)	25.8	44.3	45.6	70.9
	(10)				
DRY RESIDUE ..... lb. per hour	(11)	9.7	47.6	49.6	111.2
Char out of bottom	(12)	8.7	40.7	40.5	83.8
Dust with gas	(13)	.1	.4	.4	
Ash in total residue ..... percent	(14)	61.5	36.6		
	(15)				
GAS MADE SGC ..... Mcf per ton	(16)	77.64	45.05	43.91	28.18
Mcf per hour	(17)	1.51	2.37	2.39	2.82
B.t.u. per cu. ft. observed, gross	(18)	289	308	305	
B.t.u. per cu. ft. calculated, net	(19)	257	275	272	286
Specific gravity, calculated	(20)	.504	.509	.509	.514
Ratio H_2:CO	(21)	2.36	2.47	2.44	3.17
	(22)				
STEAM USED ..... lb. per hour	(23)				
With coal	(24)	48.5	64.4	64.4	50.6
In char zone	(25)	35.2	50.0	50.0	90.0
Undecomposed steam ..... lb. per Mcf	(26)	27.6	25.2		25.5
	(27)				
HEATING-SYSTEM DATA	(28)				
Net B.t.u. used per cu. ft. gas made ..	(29)	204	172	169	169
Heat released .... M B.t.u. per cu. ft.	(30)	16.85	22.24	22.00	26.10
Make gas used ..... Mcf per hour	(31)	1.20	1.48	1.48	1.67
CO_2 in Poc ..... percent	(32)	15.9	16.0	16.5	15.7
Primary air ..... Mcf per hour	(33)	3.11	3.92	3.85	4.53
Poc recirculated ..... do.	(34)	6.13	5.64	5.64	4.63
	(35)				
TEMPERATURES ..... °F.	(36)				
Average combustion chamber	(37)	1,775	1,805	1,810	1,810
Bottom of comb. chamber No. 1	(38)	1,910	2,000	1,995	2,025
Middle of comb. chamber No. 2	(39)	1,915	1,970	1,985	1,965
Top of comb. chamber No. 3	(40)	1,710	1,660	1,675	1,645
Outlet from comb. chamber No. 4	(41)	1,570	1,590	1,585	1,605
Inlet to fan 5	(42)	960	995	970	1,010
Air and Poc to recuperator 7	(43)	575	530	525	465
Air and Poc to furnace 8	(44)	1,230	1,305	1,260	1,330
Gas leaving retort 12	(45)	990	975	985	940
Stack 5a	(46)	815	820	825	840
Gas junction in furnace	(47)	1,690	1,605	1,605	1,420
Char zone opposite lower burners	(48)	1,605	1,535	1,575	1,425
	(49)				

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TABLE 26. - Summary data on gasification of Dakota Star Lignite in the Grand Forks, N.Dak., pilot plant

RUN AND PERIOD NUMBER		5-A	5-B	5-C	5-D
Date, December 1946	(2)	16-18	20-22	23-25	26-27
Duration ..... hours	(3)	52.75	45.83	49.25	23.92
	(4)				
COAL CHARGED ..... lb. per hour	(5)	380	323	295	380
Moisture as charged ..... percent	(6)	37.2	37.6	38.4	37.2
Ash as charged ..... do.	(7)	5.7	5.7	5.6	5.7
Gasified ..... do.	(8)	55.0	71.5	69.7	56.4
Coal per Mcf of gas ..... lb.	(9)	54.8	41.5	41.6	53.0
	(10)				
DRY RESIDUE ..... lb. per hour	(11)	71.1	44.3	35.9	72.5
Char out of bottom	(12)	68.5	42.3	31.6	68.8
Blown over at gas offtake	(13)	1.7	1.6	3.9	2.5
Dust with gas	(14)	.9	.4	.4	1.2
Ash in total residue ..... percent	(15)	23.7	26.2	30.2	20.6
	(16)				
GAS MADE SGC ..... Mcf per ton	(17)	36.53	47.97	48.00	37.68
Mcf per hour	(18)	6.94	7.79	7.09	7.17
B.t.u. per cu. ft. observed, gross	(19)	272	266	257	270
B.t.u. per cu. ft. calculated, net	(20)	236	237	231	238
Specific gravity, calculated	(21)	.541	.542	.545	.549
Ratio H_2:CO	(22)	5.24	7.18	7.00	5.04
	(23)				
STEAM USED ..... lb. per hour	(24)				
With coal	(25)	147	243	220	145
In char zone	(26)	235	260	395	245
Undecomposed steam ..... lb. per Mcf	(27)	53.6	57.0	76.4	52.5
	(28)				
HEATING-SYSTEM DATA	(29)				
Net B.t.u. used per cu. ft. gas made...	(30)	139	126	144	140
Heat released ..... M B.t.u. per cu.ft.	(31)	8.41	8.55	8.86	8.75
Make gas used ..... Mcf per hour	(32)	4.10	4.33	4.42	4.42
CO_2 in Poc ..... percent	(33)	17.4			
Primary air ..... Mcf per hour	(34)	9.52	9.61	9.73	9.04
Poc recirculated .....do.	(35)	36.97	38.36	38.02	38.49
	(36)				
TEMPERATURES ..... °F.	(37)				
Average combustion chamber	(38)	1,500	1,510	1,535	1,535
Bottom of comb. chamber No. 1	(39)	1,775	1,750	1,750	1,770
Middle of comb. chamber No. 2	(40)	1,610	1,620	1,650	1,650
Top of comb. chamber No. 3	(41)	1,375	1,395	1,430	1,420
Outlet from comb. chamber No. 4	(42)	1,250	1,270	1,305	1,300
Inlet to fan 5	(43)	700	690	700	700
Air and Poc to recuperator 7	(44)	520	520	530	540
Air and Poc to furnace 8	(45)	995	1,010	1,030	1,025
Gas leaving retort 12	(46)	600	655	650	605
Stack 5a	(47)	495	480	490	495
Steam to char zone	(48)	250	275		250
Steam with coal	(49)	480	475	485	480
	(50)				

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TABLE 27. - Proximate and ultimate analyses of coals used for runs 13, 14 and 15 at Golden, Colo., and for run 5 at Grand Forks, N. Dak.

Description	Run and period	Condition[1]/	Pittsburgh lab. No.	Proximate, percent				Ultimate, percent						B.t.u. per pound	Softening temp. ash, °F.
				Moisture	Volatile matter	Fixed carbon	Ash	Hydrogen	Carbon	Nitrogen	Oxygen	Sulfur			
Colorado subbituminous B Caprock mine	13-A and C	1	C-63134	22.1	32.5	39.8	5.6	6.0	53.2	0.7	34.0	0.5		9,150	2,050
		2			41.8	51.0	7.2	4.6	68.3	.9	18.3	.7		11,750	
		3			45.0	55.0		4.9	73.6	.9	19.9	.7		12,660	
Colorado subbituminous B Caprock mine	15-A and C	1	C-69508	21.5	33.8	39.3	5.4	6.0	54.1	.7	33.1	.7		9,250	2,060
		2			43.0	50.1	6.9	4.6	69.0	.9	17.7	.9		11,790	
		3			46.2	53.8		4.9	74.1	1.0	19.1	.9		12,660	
Hyderabad-Deccan, India subbituminous A	14-A, B, C and D	1	C-63135	11.0	27.3	45.2	16.5	4.8	58.5	1.2	18.3	.7		10,020	2,610
		2			30.7	50.7	18.6	4.0	65.7	1.4	9.5	.8		11,260	
		3			37.7	62.3		4.9	80.6	1.7	11.8	1.0		13,820	
North Dakota lignite Dakota Star mine	5-A, B, C and D	1	C-67425	36.2	27.3	30.1	6.4	6.7	41.4	.6	44.2	.7		6,960	2,310
		2			42.7	47.3	10.0	4.2	64.8	1.0	18.8	1.2		10,910	
		3			47.5	52.5		4.6	72.0	1.1	21.0	1.3		12,110	

1/ Condition: (1) As received; (2) moisture-free; (3) moisture- and ash-free.

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TABLE 28. - Analyses of gases from Colorado subbituminous B coal, subbituminous A coal from Hyderabad-Deccan, India, and North Dakota lignite1/

Run and period	Analysis, percent								Specific gravity (air = 1.0)	Gross B.t.u. per cu. ft.	
	CO_2	Ill.2/	O_2	CO	H_2	CH_4	C_2H_6	N_2	calculated	Obs.	Calc.
13-A	12.2	0.4	0.0	28.2	55.6	3.3	0.0	0.3	0.523	309	307
13-C	12.5	.3	.0	27.8	56.0	3.3	.0	.1	.522	307	307
15-A	12.7	.2	.0	27.3	56.4	2.9	.0	.5	.521	297	300
15-C	17.4	.4	.0	21.6	57.0	2.8	.0	.8	.545	288	287
14-A	13.4	.2	.0	25.1	59.1	1.8	.1	.3	.505	289	292
14-B	13.4	.6	.0	23.8	58.5	3.0	.3	.4	.516	308	310
14-C	13.3	.6	.0	23.9	58.3	3.0	.3	.6	.509	305	309
14-D	15.3	.9	.0	20.9	58.4	4.3	.0	.2	.515		314
5-A	23.6	.2	.0	11.7	61.3	2.4	.6	.2	.541	273	273
5-B	24.8	.2	.0	8.6	61.6	3.2	.6	1.0	.542	266	271
5-C	25.4	.2	.0	8.8	62.0	2.3	.6	.7	.545	257	265
5-D	23.6	.2	.0	12.0	60.1	3.3	.3	.5	.548	270	272

1/ Runs 13-A, 13-C, 15-A and 15-C made on Colorado subbituminous B coal - Golden pilot plant; runs 14-A, 14-B, 14-C and 14-D made on subbituminous A coal from Hyderabad-Deccan, India - Golden pilot plant; runs 5-A, 5-B, 5-C and 5-D made on North Dakota lignite - Grand Forks pilot plant.

2/ Illuminants as $C_{2.8}H_{5.6}$.

The development of the annular retort had required many changes in the pilot plants to find the best arrangement of the reaction zone and to determine the optimum width of the annulus. Much of this work has been done with mild-steel retorts because it was necessary to make changes to study new conditions. It is now indicated that the best arrangement of the retort assembly for the large plant is that illustrated in figure 42, which incorporates suitable alloy parts throughout. Materials to fabricate this assembly were ordered during 1947 and will be installed and tested during 1948. A cast-alloy retort having a 3/4-inch wall thickness and a composition of 25 percent chromium and 20 percent nickel has been procured for testing. The inner retort parts will be made of type 430 alloy having about 17 percent chromium, and the recuperator will be fabricated from type 309 alloy containing 25 percent chromium and 12 percent nickel.

During the year it was demonstrated in the small pilot plant that heating the retort to a maximum temperature of 1,900° F. resulted in a heat transfer of approximately 6,000 B.t.u. per hour per square foot and a production of 60 cubic feet of ($CO + H_2$) per hour per square foot. This important demonstration clearly indicates that the cost of the alloy for 10,000 hours of service will be about 1 cent per thousand cubic feet of ($CO + H_2$) or approximately one-fourth the cost of oxygen required to make the same quantity of gas.

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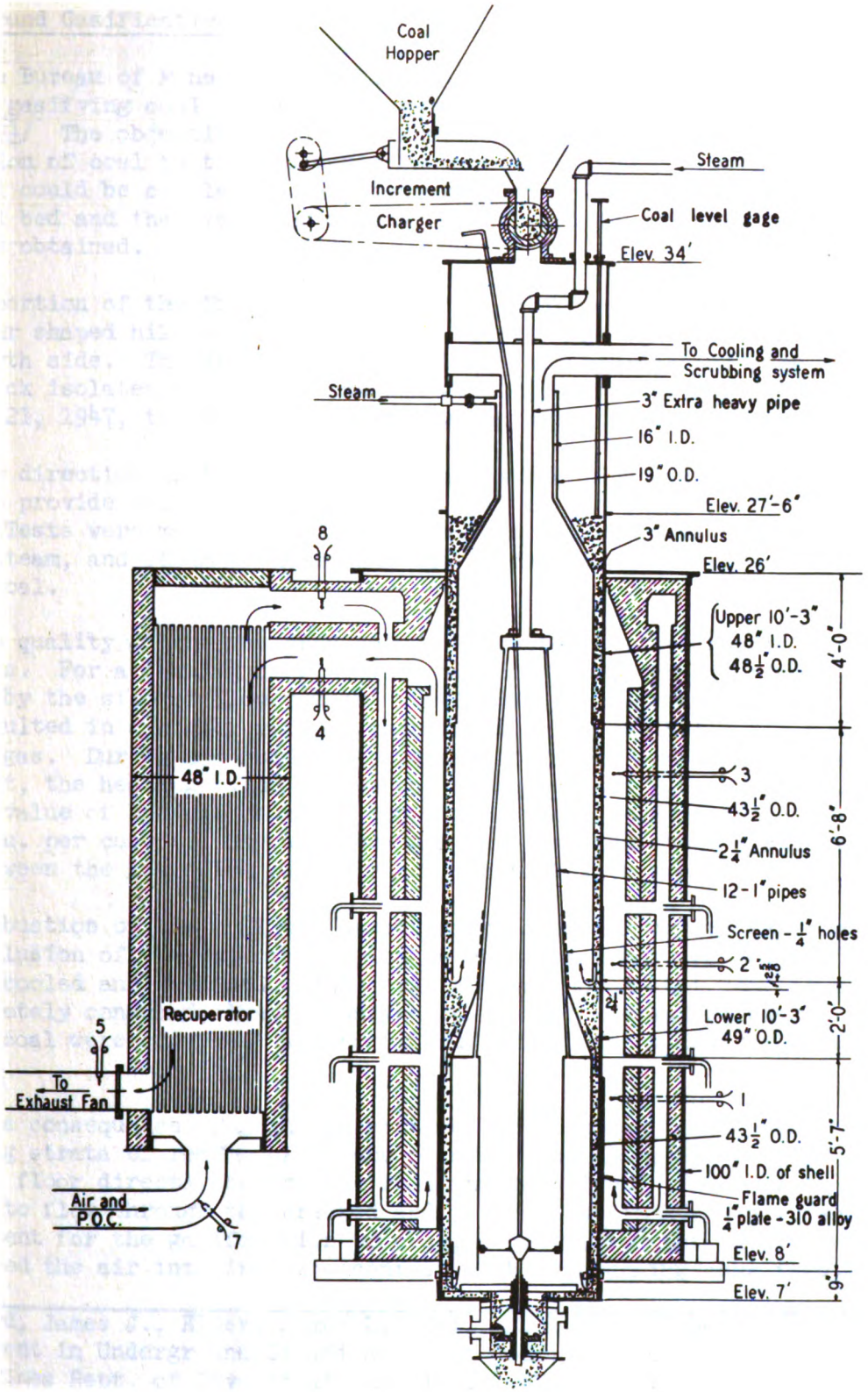

Figure 42. - Proposed arrangement of retort assembly for Grand Forks, N. Dak., pilot plant.

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Underground Gasification

The Bureau of Mines and the Alabama Power Co. cooperated in an experiment in gasifying coal in place underground, at Gorgas, Walker County, Ala.60,61/ The objectives of this experiment were to determine whether combustion of coal in the bed could be maintained and controlled, whether the coal could be completely gasified, what effect the fire would have on the coal bed and the overlying strata, and the yield and quality of gas that could be obtained.

A portion of the Pratt coal bed which outcropped on three sides of an irregular shaped hill was isolated by a cut through the hill and coal bed on the fourth side. The development of a U-shaped experimental mine in the 35-inch-thick isolated portion of the bed was begun in November 1946, and on January 21, 1947, the mine was ignited with thermite bombs.

The direction of flow of air and gas in the mine was reversed periodically to provide uniform burning of the coal in the pillar between the entries. Tests were made with air, oxygen-enriched air, air-oxygen-steam, oxygen-steam, and steam alone during the 50-day period of forced burning of the coal.

The quality of the gas produced was affected by the physical conditions at Gorgas. For a burning face as long as that used, the air flow, which was limited by the size of blower used, should have been greater. This would have resulted in a higher rate of combustion, a longer hot zone, and better-quality gas. During a considerable period of fairly stable operation with air blast, the heating value of the gas averaged 46.8 B.t.u. per cu. ft. Heating value of the gas in short runs with steam averaged approximately 200 B.t.u. per cu. ft.; and with the air, oxygen, and steam mixtures averaged between the low value with air and the high with steam.

Combustion of the coal was easily maintained during the experiment. At the conclusion of the experiment, active combustion was stopped and the residue was cooled and examined. This examination showed that coal in place can be completely consumed, leaving only a residue of ash and clinker. Some 236 tons of coal were consumed, and no islands of unreacted coal or coke were found.

As a consequence of high temperatures developed at the burning face, the overlying strata of roof rock became plastic, expanded, and settled down on the mine floor directly behind the reacting coke face. This forced the air and gas to flow through the cracked and porous coke face, a very favorable development for the gasification process, as it filled the burned-out area and forced the air into intimate contact with the burning coke face.

60/ Dowd, James J., Elder, James L., Capp, J. P., and Cohen, Paul, Experiment in Underground Gasification of Coal, Gorgas, Ala.: Bureau of Mines Rept. of Investigations 4164, 1947, 62 pp.

61/ McCabe, Louis C., Gasification of Coal Underground: Min. Cong. Jour., vol. 33, No. 8, August 1947, pp. 42-45.

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This behavior of the cap rock confirmed a preliminary test made on a slab of the rock removed from the mine before firing and shipped to Pittsburgh for laboratory testing over a gas-fired test furnace. The similar behavior of the cap rock in the laboratory test and in the mine indicates that such preliminary laboratory tests may provide valuable prior evidence of conditions to be expected in a mine during underground gasification.

LIQUEFACTION

A report of Bureau of Mines progress in the Synthetic Liquid-Fuels Program has been given to Congress in accordance with the act authorizing the program.62/

The authorization and objectives of the Synthetic Liquid-Fuels Program and the need for synthetic fuels, including raw materials and methods of converting, with a discussion of European developments and their application to American conditions, have been discussed.63/ The laboratories and demonstration plants and progress for their operation and the division of duties within the Bureau of Mines are given in that same publication.

In response to a request for information written in popular style, an article describing the production of gas for synthetic fuel, the synthesis of oil from this gas, and known plans for commercial development was issued.64/

Facilities for Research and Development, Bruceton, Pa.

The buildings, including all services, are almost completed. An airplane view of the buildings is shown in figure 43. The center building, which houses the administrative offices, drafting room, instrument shop, store rooms, and cafeteria, was completed in May 1947. Occupation of the coal-hydrogenation building, which is the main building shown at the right of the photograph, was started in April 1947, when the experimental plant at Pittsburgh was dismantled and moved to Bruceton. The large building shown at the left of the group of main buildings houses the Fischer-Tropsch research and demonstration laboratories. The power plant and coal-washing laboratory are shown in the foreground of the picture immediately adjacent to the railroad siding. The sewage-disposal plant appears in the foreground of the picture, and the smaller buildings to the right of the main buildings are provided for oil storage and for the preparation of hydrogenation and carbon monoxide.

62/ Secretary of the Interior, Report on the Synthetic Liquid Fuel Act from January 1, 1946 to December 31, 1946: 82 pp.

63/ Fieldner, A. C., Synthetic Liquid Fuels Investigations of the Bureau of Mines: Science Counselor, March 1947, vol. 10, No. 1, pp. 8-10, 24-26.

64/ Newman, L. L., Synthetic Oil: Federal Sci. Prog., February 1947, pp. 11-13.

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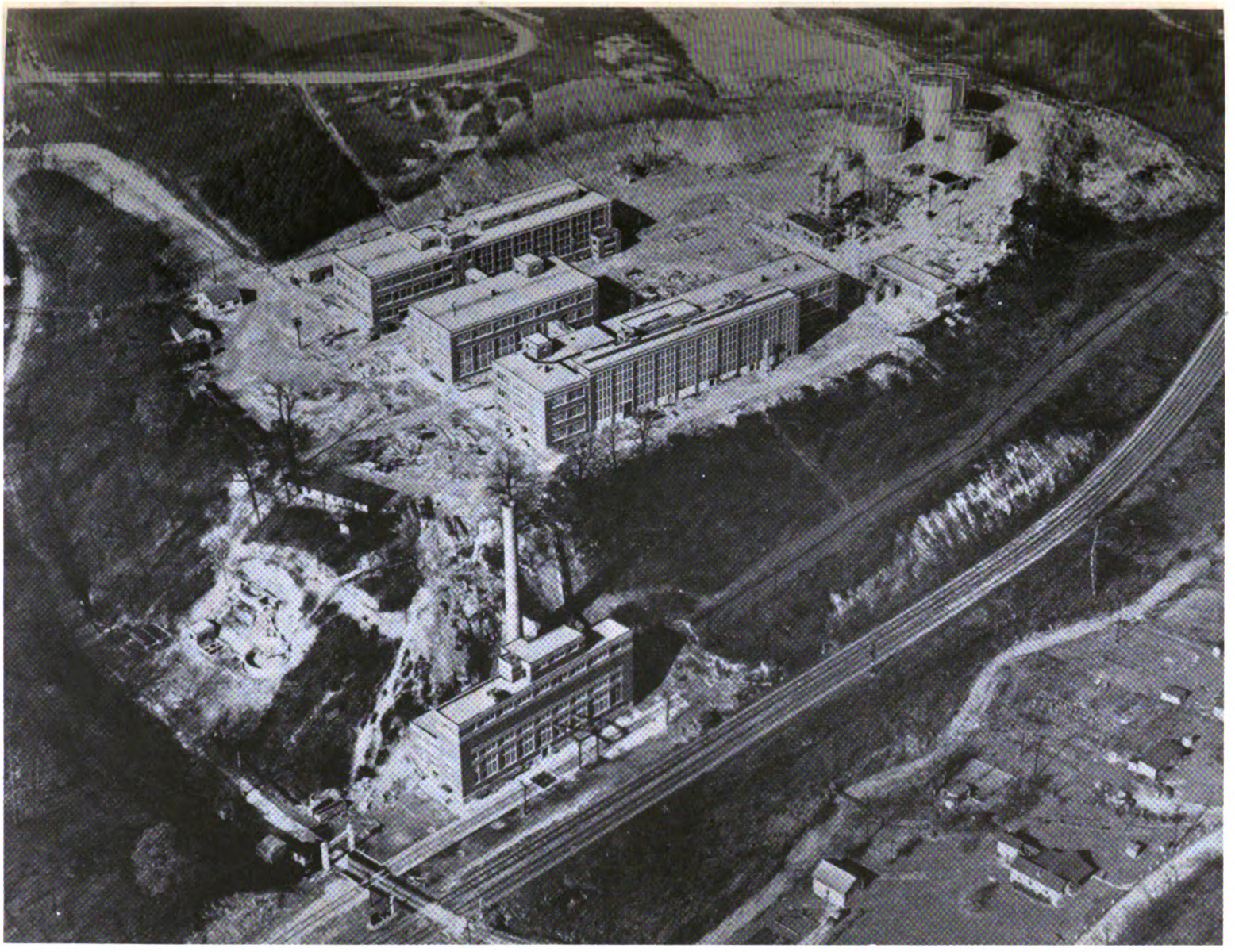

Figure 43. - Airplane view of synthetic liquid fuel laboratory and pilot-plant buildings at Bruceton, Pa.

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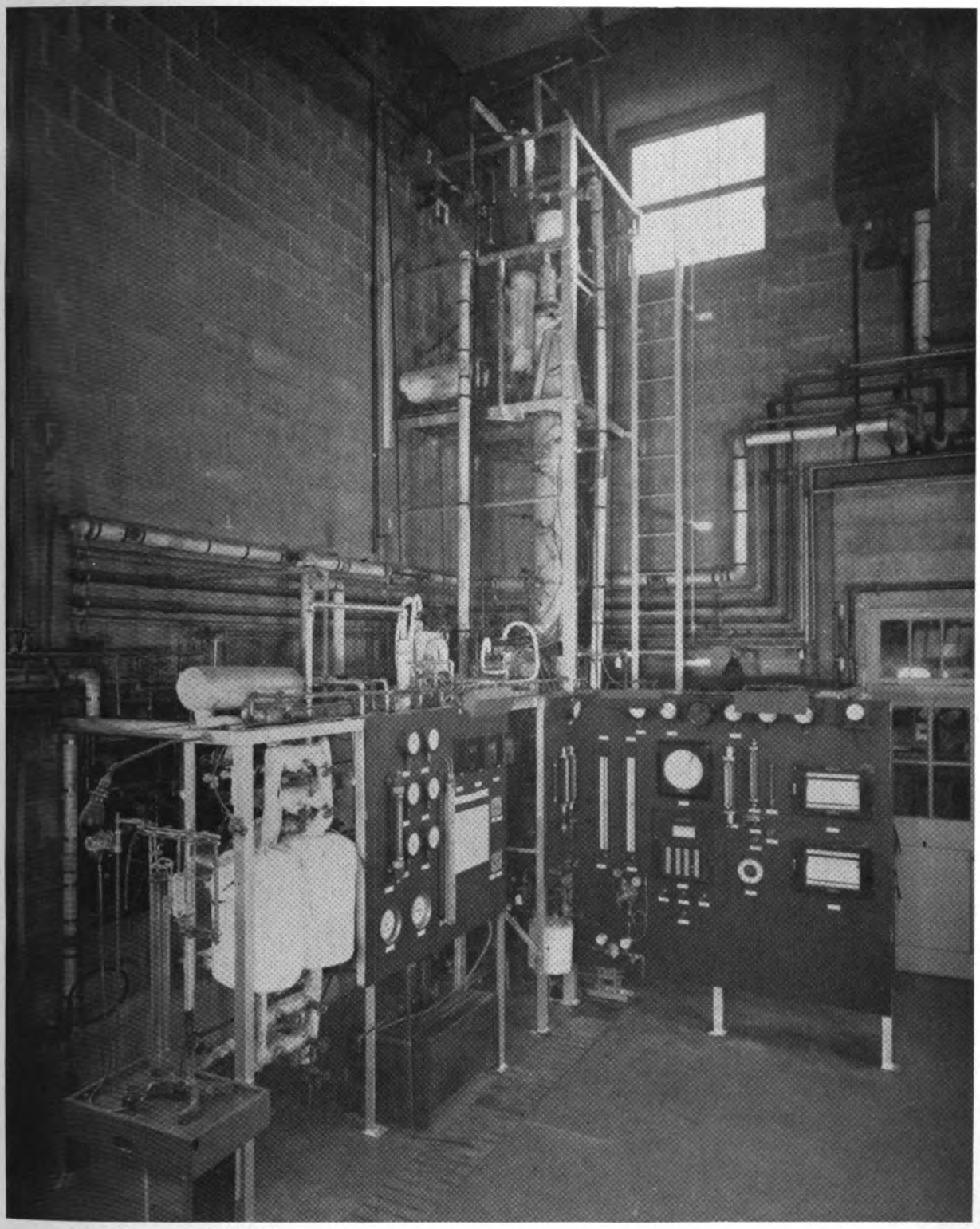

Figure 44. - Evaporative cooling pilot plant for synthesis of liquid fuel by hydrogenation of carbon monoxide.

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Synthesis of Liquid Fuels from Hydrogen and Carbon Monoxide

Process Development; Pilot-Plant and Bench-Scale Tests

The preliminary work on the use of oil evaporation as cooling medium for the Fischer-Tropsch synthesis was described in last year's report.65/ The redesigned, internally cooled converter (by oil evaporation), built to accommodate a catalyst bed 3 inches in diameter by 8 feet deep, is shown in figure 44. Adequate control of all operating conditions such as pressure, temperature, rate of cooling oil circulation, space velocity, and rate of recycling of end gas was provided in the unit. In addition, provision was made for sample withdrawal and cooling oil injection at several points in the 8-foot column. About eight operations of 100 to 200 hours duration have been conducted with pelleted cobalt-thoria-Filter Cel catalyst since the unit was completed.

Experiments with countercurrent flow of cooling oil showed that the maximum space velocity (volumes of synthesis gas per volume of catalyst per hour) permissible without flooding is about 200; tests with cocurrent flow indicated that space velocities of 600 or greater could be used without flooding. The weight of oil obtained per unit volume of catalyst per day (space-time yield) increased two and one-half fold when the space velocity was increased from 100 to 300. The gaseous hydrocarbons, methane and ethane, constituted only 15 to 20 percent of the hydrocarbon product. The space-time yield increased three and one-half fold when the space velocity was increased from 100 to 450. A space velocity of 600 resulted in only a slight further increase in space-time yield.

The results thus far obtained indicate that it will be possible to obtain 3 to 5 times the weight of oil per unit volume of catalyst per day that is obtained in the Germany Ruhrchemie process, and it shows promise of equaling in industrial applicability the fluidized catalyst process recently developed by American oil-company engineers. The internally cooled converter process is more versatile than the fluidized-catalyst process in the respect that it is not limited to the use of only those catalysts that have certain critical fluidization characteristics. Additional extended operations are necessary with different modes of cooling-oil distribution, with a wider range of space velocities and at various operating pressures. It will be necessary also to determine the effect of recycling end gases and the ultimate yield, using multistage operation.

A modification of operating procedure tested recently was the complete submersion of the catalyst in cooling oil, the synthesis gas being introduced at the bottom of the converter. This procedure promises to eliminate the major source of trouble in the operation of the internally cooled converter - that is, variation in distribution of cooling liquid in the column, which results in formation of hot spots and overheating of the catalyst.

65/ Fieldner, A. C., and Ambrose, P. M., Annual Report of Research and Technologic Work on Coal, Fiscal Year 1946: Bureau of Mines Inf. Circ. 7417, 1947, 142 pp.

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Experiments have been in progress for about 2 months using a pelleted iron-copper-potassium carbonate catalyst in the internally cooled converter. Operation with this catalyst has been much smoother than with cobalt, largely because of the wider temperature range that may be employed without excessive formation of methane.

The enlarged process development laboratory is continuing its exploratory "bench" or laboratory-size experiments on several processes. A view of this laboratory is shown in figure 45. Three processes are now under investigation. They are:

1. The liquid-phase catalyst suspension process, which was described in last year's report. The experimental converter is 3 inches in diameter with a maximum capacity of 2 liters of catalyst suspension. The operating pressure is in the range of 1 to 30 atmospheres. Porous disks fabricated of sintered iron powder are used for distributing the feed gas. Early results of the investigation of operating variables in this process show that high pressures, high concentrations of catalyst in the slurry, and high gas input favor the reaction by increasing the absorption rate of the synthesis gas. Studies of catalyst-conditioning (starting the synthesis) and reactivation procedures are in progress. Based on the results thus far obtained, a new reactor has been designed to obtain maximum gas to liquid interfacial area.

2. The hot-gas recycle process, in which all of the heat evolved during the synthesis is carried out of the synthesis vessel by the sensible heat of the gas. This process involves very large recycle rates of the order of 25 to 100 parts of recycle gas to 1 part of fresh gas. A cost estimate showed that such recycling would add about 1 cent per gallon to the cost of production, as compared with other processes in which only 2 to 5 parts of recycle gas to 1 part of fresh gas are used. Further analysis indicates that, with modification of catalyst shape and extent of recycle, the process may be economically feasible. Bench-scale equipment has been designed for some experimental work on this process.

3. Experiments on fluidized fixed-bed processes are currently directed towards a study of the factors controlling the rate of elemental carbon deposition on a fluidized iron catalyst. This problem now is of critical importance in the design of commercial plants using fluidized iron catalyst systems.

Preparation and Testing of Catalysts

X-ray diffraction patterns and magnetic susceptibility[66/] of Fischer-Tropsch catalysts were reported.

In the preparation of large batches of precipitated iron catalysts under presumably identical conditions, it was observed that some were glossy black

66/ Hofer, L. J. E., Peebles, W. C., and Dieter, W. E., X-Ray Diffraction and Magnetic Studies of Unreduced Ferric Oxide Fischer-Tropsch *Catalysts*: Jour. Am. Chem. Soc., vol. 68, 1946, pp. 1953-1956.

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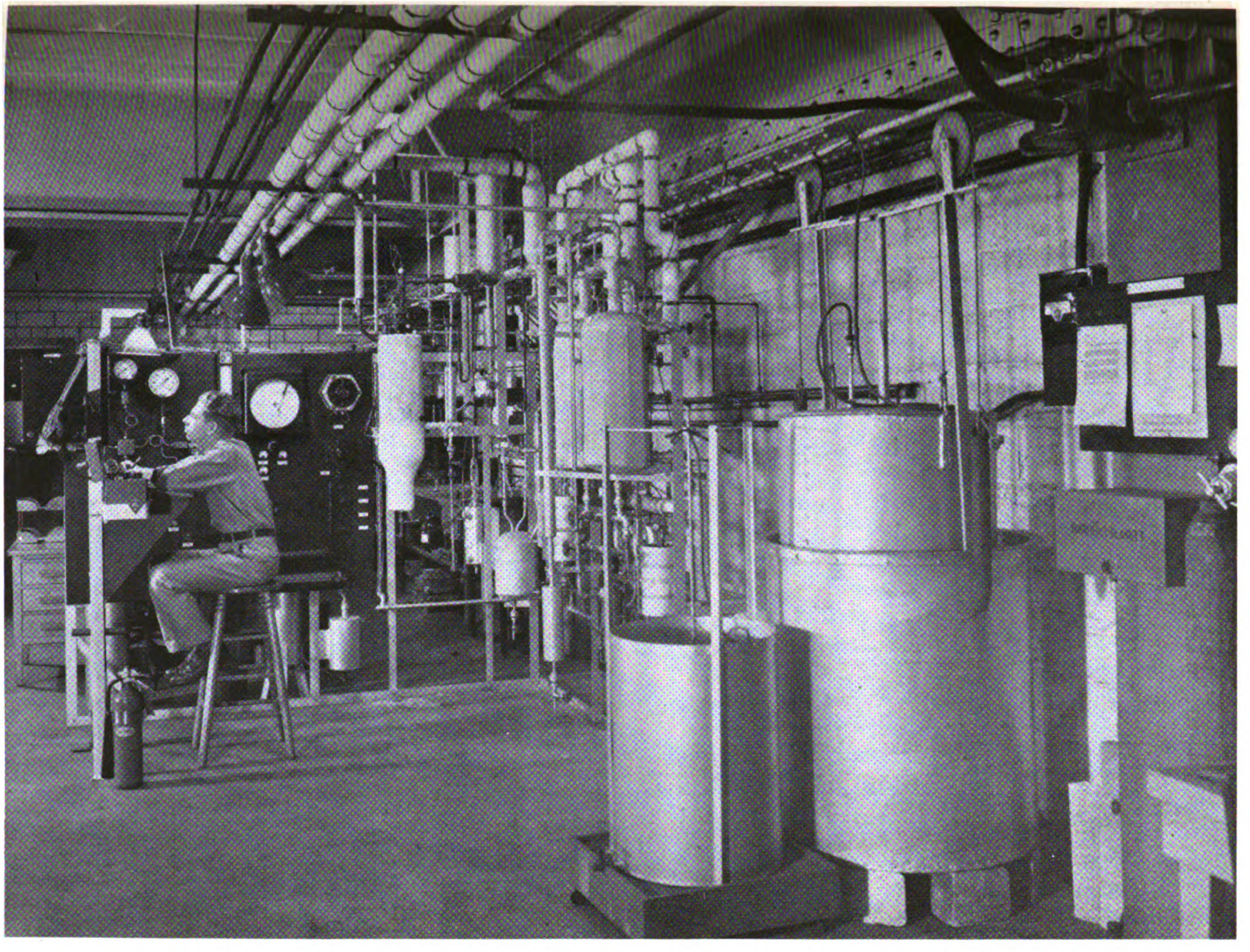

Figure 45. - Bench-scale unit for liquid-phase process for synthesis of liquid fuel by hydrogenation of carbon monoxide.

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and others were brown and much less shiny. Surface areas of these preparations were measured by determining the nitrogen absorption isotherms at -195° C. of catalyst in the unreduced state and after induction at 230° C. in $1H_2 + 1CO$ gas mixture. Chemisorption of carbon monoxide at -195° C. was measured to determine the fraction of metallic iron present on the catalyst surface. From the data shown in table 29, it was observed that both catalysts had a high surface area and small pore structure in the unreduced form. The isotherm of the black form of the catalyst was of the type observed in silica gels, but the brown form was unusual in being linear from a relative pressure of 0.1 to 0.7. When the catalysts were inducted for 2 hours at 1 atmosphere pressure and 230° C. with $1H_2 + 1CO$ gas, the areas dropped to 22.9 to 15.5 square meters per gram for the black and brown catalysts, respectively. These are consistent with other data upon the change in surface areas of gel-type iron catalysts.

TABLE 29. - Properties of $Fe-Cu-K_2CO_3$ catalysts

Catalyst			Unreduced		After induction			
No.	Appearance	Percent iron	Surface area $m.^2/g.$	Average pore diameter, Å 1/	Weight loss, percent	Surface area $m.^2/g.$ 2/	V_{CO} chemi cc./g. 2/	$V_{Co\ chemi}/V_m\ N_2$ 3/
Composite P-3003.03 P-3003.042 P-3003.05	(black)	56.7	303	28.0	10.3	22.9	0.93	0.18
P-3003.07	(brown)	59.5	135	41.6	5.9	15.5	.70	.20

1/ Calculated by the formula $d = \frac{4\ V_1}{A}$, where A is the surface area and V_1 the volume adsorbed at 0.98 relative pressure computed as liquid.
2/ Per gram of unreduced catalyst.
3/ V_{CO} chemi = volume of chemisorbed CO, V_m = cc. of N_2 in monolayer.

Dr. H. Pichler, after a visit in May 1947 to the Kaiser Wilhelm Institute, reports the results of a recent pilot-plant test made in one tube of the middle-pressure reactor using a catalyst prepared from a mixture of ferrous and ferric chlorides plus some copper as promoter. The catalyst was inducted by treatment with $1H_2 + 1CO$ gas at about 225° C. for 48 hours. Operation was then started at 10 atmospheres and 200° C. This catalyst was still at peak activity after 4-1/2 months of continuous operation; the present operating temperature is 205° C.

Data were obtained on various physical properties of unreduced-and reduced-cobalt catalysts and of different types of diatomaceous earth (kieselguhr) - namely, X-ray diffraction patterns, surface areas, and porosity.67/

67/ Weller, S., Chemisorption of Gases: Jour. Chem. Phys., vol. 15, 1947, p. 336.

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X-ray diffraction patterns of natural diatomaceous earths and of unreduced cobalt-thoria-natural diatomaceous earth catalysts showed them to be almost completely amorphous, the sharpest line in the pattern being that due to the quartz present in the diatomaceous earth; the pattern of alkali-treated and calcined diatomaceous earth showed a well-developed crystal structure. The X-ray pattern of reduced cobalt-thoria-kieselguhr and cobalt-thoria-magnesia-kieselguhr catalysts showed the lined of cubic cobalt crystals. As the cubic form is unstable at room temperature, it is likely that the thoria and magnesia have an inhibiting effect on the conversion of cubic to hexagonal cobalt.

The total surface area of reduced-and unreduced-cobalt catalysts was measured by determining the nitrogen adsorption isotherms at -195° C. After reduction of the catalyst, the fraction of metallic cobalt in the surface was estimated by determining the carbon monoxide isothern at -195° C. Figure 46 shows the nitrogen isotherms of kieselguhr and of an unreduced cobalt-thoria-magnesia-kieselguhr catalyst, and figure 47 shows the nitrogen adsorption isotherms for unreduced and reduced samples of the same catalyst. The isotherms are of the usual S-shaped type, with only a slight amount of hysteresis, indicating that the pore structure consists of capillaries, most of which are larger than 500 Ångstrom units in diameter. The nitrogen and carbon monoxide isotherms lie one above the other, the difference between them representing the quantity of chemisorbed carbon monoxide. Most of the area in the unreduced catalyst is provided by the cobalt oxide promoter-complex and only little by the kieselguhr. The surface areas of the catalysts are not additive with respect to the areas of the kieselguhrs used. Catalysts prepared from alkali-treated and calcined kieselguhr have lower areas than those prepared from natural kieselguhr; and in most cases the difference is greater than can be accounted for by assuming additivity of surface areas of the kieselguhr and the cobalt-promoter complex. Surface area measurements on cobalt-thoria-magnesia-diatomaceous earth catalysts showed that their activity in the synthesis varies directly with the surface area of the unreduced catalysts.

From the data shown in table 30, it was observed that upon reduction in a rapid stream of hydrogen at 400° C. for 2 hours, the surface area of the doubly promoted cobalt-thoria-magnesia-natural kieselguhr decreased to 75 percent of that of the unreduced catalyst, whereas the surface area of the singly promoted cobalt-thoria-natural kieselguhr catalyst decreased to 35 percent of that of the unreduced catalyst. Thus, the presence of promoters in cobalt catalysts makes reduction more difficult and prevents sintering. It is of interest to note that the surface area of the doubly promoted catalyst containing alkali-treated and calcined kieselguhr decreased to 55 percent of that of the unreduced catalyst.

Kinetic studies of rates of carbiding of cobalt and iron catalysts by carbon monoxide, of hydrogenation of the carbide, and of the synthesis have yielded some information concerning the temperature and pressure dependence of these rates. A general reaction-rate law, which was not restricted to constant pressure or constant volume and which was applicable to flowing

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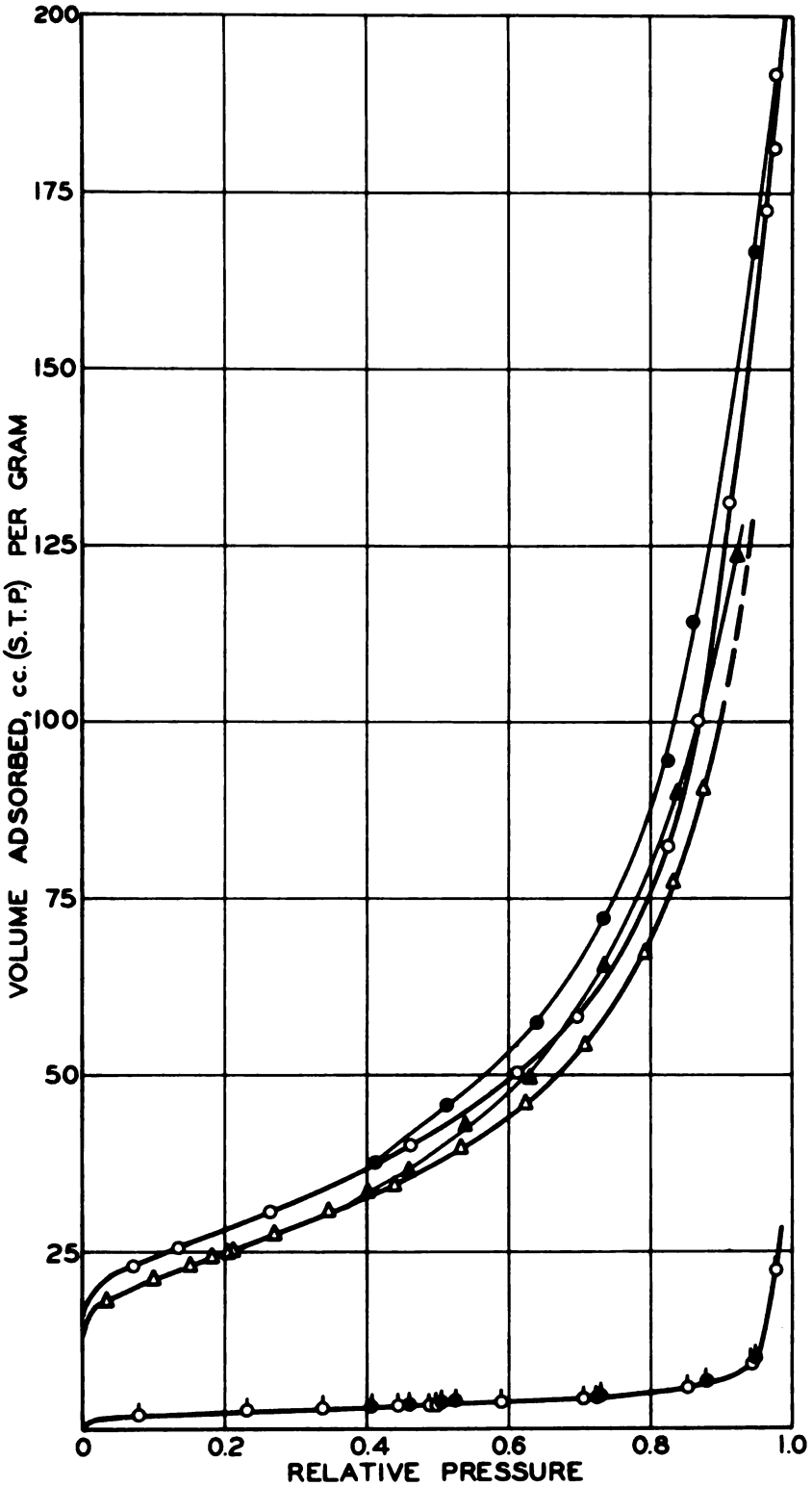

Figure 46. - Adsorption isotherms of nitrogen at -195° C. of unreduced cobalt Fischer-Tropsch catalyst 89J in granular and pelleted forms and kieselguhr used as catalyst support plotted for weight of kieselguhr per gram of catalyst. O represents 89J granules; Δ represents 89J pellets; and ȯ represents kieselguhr. Adsorption points are open and desorption points solid.

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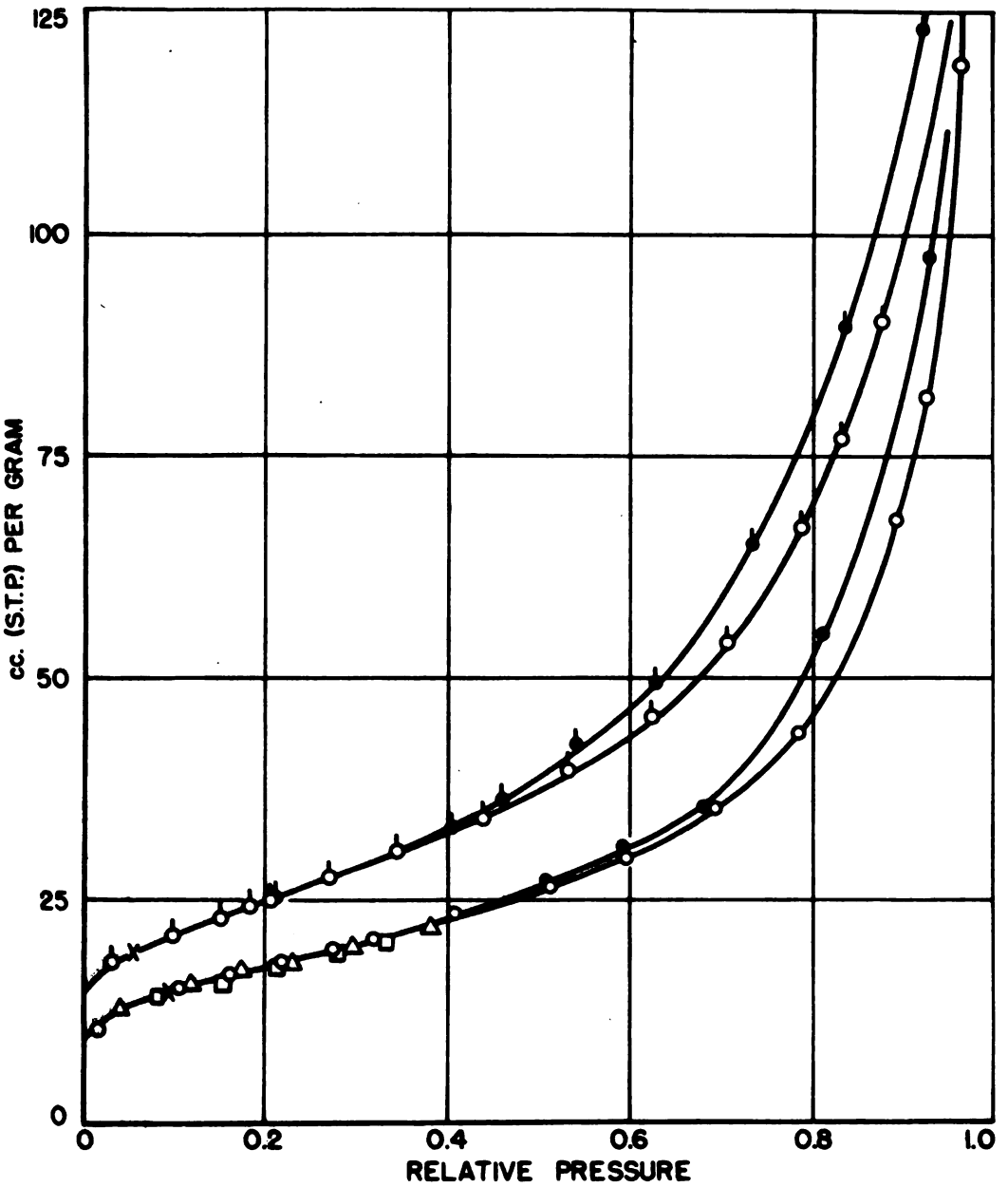

Figure 47. - Adsorption of nitrogen at -195° C. on unreduced- and reduced-cobalt catalyst 89J. ◊ represents unreduced catalyst, and ○, □, and △ represent different samples of reduced catalyst. Desorption points are solid, and volume of gas corresponding to a monolayer is represented by X.

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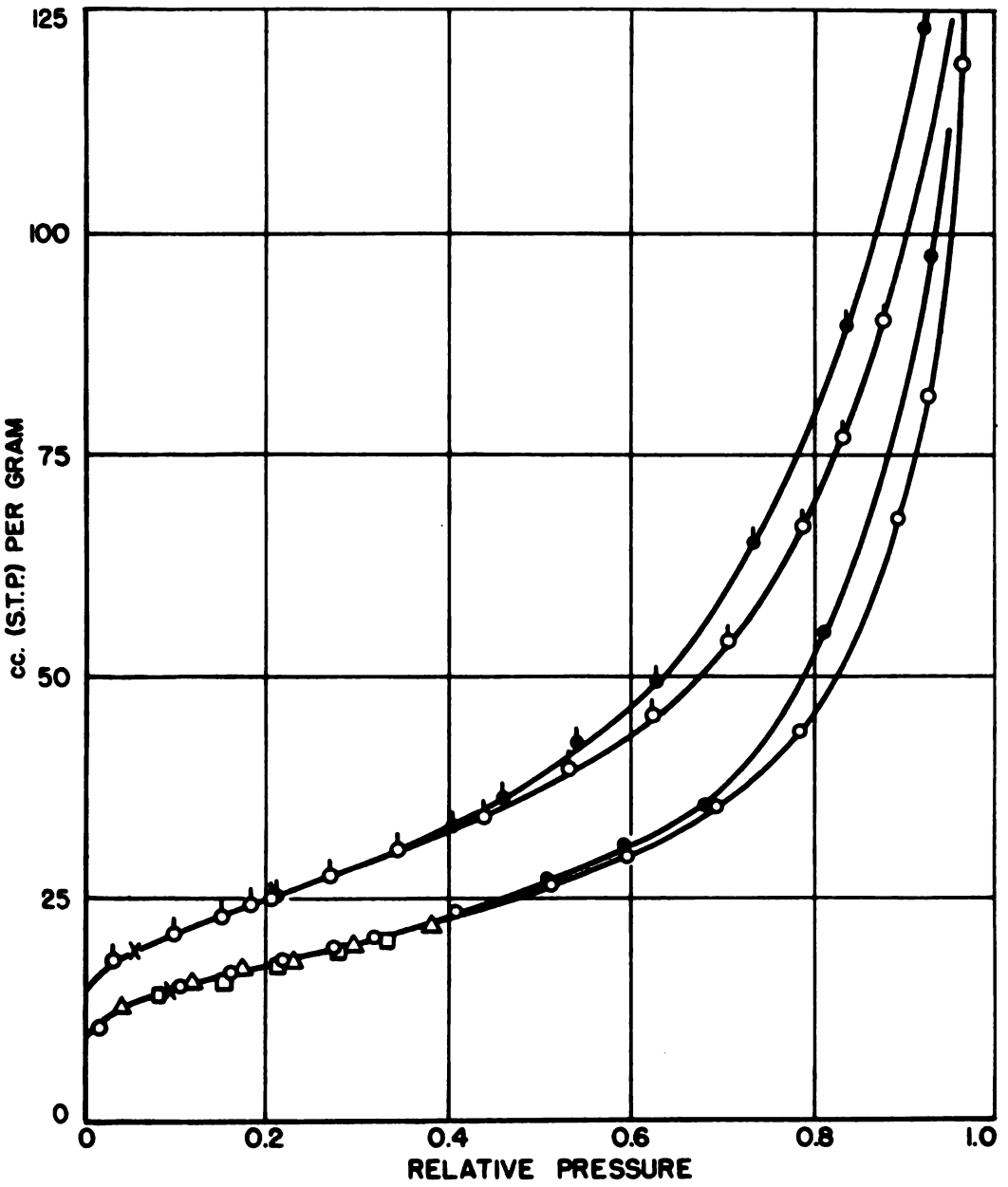

Figure 47. - Adsorption of nitrogen at -195° C. on unreduced- and reduced-cobalt catalyst 89J. ◊ represents unreduced catalyst, and ○, □, and △ represent different samples of reduced catalyst. Desorption points are solid, and volume of gas corresponding to a monolayer is represented by X.

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systems, was obtained for homogeneous isothermal reactions in ideal systems.[68] Mention was made of modifications required for the consideration of systems that are nonisothermal. These results will be of value in the interpretation of data obtained experimentally in kinetic studies of the mechanism of the Fischer-Tropsch reaction.

TABLE 30. - Adsorption of nitrogen and carbon monoxide at -195° C. on unreduced- and reduced-cobalt catalysts and cobalt oxide

Catalyst	Reduction method			Loss, weight percent	Surface area, $m.^2/g.$[2]	V_m nitrogen, cc./g.[2]	V_{chemi} carbon monoxide, cc./g.[2]
	Temperature, °C.	Hours	Space velocity/hour[1]				
89H[3]	-	-	-	0.0	67.2	15.4	0.0
89H	400	2	6,000	17.9	36.9	8.4	4.3
89J[3]	-	-	-	.0	84.1	-	.0
89J	400	2	6,000	17.7	62.0	14.2	3.1
108B[4]	-	-	-	.0	84.6	19.3	.0
108B	400	2	6,000	28.6	28.3	6.5	-
Co_3O_4	-	-	-	.0	68.0	15.4	.0
Co_3O_4	250	41	100	30.6	3.2	.74	.48

1/ Volumes hydrogen per bulk volume of catalyst per hour.
2/ All adsorption data per gram of unreduced catalyst.
3/ Cobalt-thoria-magnesia-kieselguhr.
4/ Cobalt-thoria-kieselguhr.

Rates of carbiding by carbon monoxide, of hydrogenation of the carbide, and of hydrocarbon synthesis from $2H_2 + 1CO$ gas mixture were measured for a cobalt-thoria-kieselguhr and a cobalt-thoria-magnesia-kieselguhr catalyst using an all glass apparatus, including a circulating pump. A similar investigation was begun for a precipitated iron catalyst. The gases were circulated over the catalyst and then through the liquid air traps so as to condense all products except about 1 cm. partial pressure of methane. The pressure was automatically adjusted to a constant value during each experiment, and the change in volume of the reactants was measured. It was found that the initial carbiding rate for cobalt catalysts was of the same order of magnitude as the synthesis rate, but that the subsequent "steady-state" rate of carbiding of the bulk of the catalyst was about ten times lower than the rate of the synthesis. The rate of hydrogenation of the carbide was very much faster than carbiding and synthesis at synthesis temperatures. Per unit of metal surface, carbiding of the iron catalyst proceeded about five times more quickly than the carbiding of the cobalt catalyst. However, hydrogenation of iron carbide proceeded more slowly than carbiding, at synthesis temperature. It was observed that the temperature and pressure dependence of the carbiding rate were essentially the same for cobalt and iron catalysts. The implication is that the same step is rate-determining for the carbiding of both metals.

68/ Brinkley, S. R., Jr., and Weller, S., Generalized Form of the Reaction-Rate Law for Homogeneous Reactions: Jour. Am. Chem. Soc., vol. 69, 1947, pp. 1319-1321.

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An investigation of the role of cobalt and iron carbide in the Fischer-Tropsch synthesis is under way because the theory has long been popular that during the reaction these metal carbides are formed and subsequently reduced to methylene (CH_2) groups. The concept of metal carbide as a synthesis intermediate has been used rather loosely. Distinction has rarely been made between bulk carbide and "surface" carbide as a possible intermediate. The idea of "surface" carbide is elusive, since it is very difficult to distinguish between a "surface" carbide and adsorbed carbon monoxide. However, evidence has been accumulated which demonstrates that bulk carbide, in the case of cobalt catalysts, probably is neither an intermediate in the synthesis nor a catalytically active substrate for the synthesis.69/

Recent work in the physical chemistry and catalyst-testing laboratories shows that the presence of extensive amounts of carbide in cobalt catalysts severely inhibits the Fischer-Tropsch synthesis. In one experiment, for example, a cobalt-thoria-magnesia-kieselguhr (100:6:12:200) catalyst was reduced with hydrogen at 400° C. and carbided with carbon monoxide for 20 hours at 200° C.; the volume of carbon monoxide reacted was equivalent to 70 percent conversion of the cobalt to Co_2C. Exposure of this carbided sample to circulating synthesis gas resulted in an over-all synthesis rate at 150° C. of 60-70 x 10^{-4} grams of carbon per gram of cobalt per hour. After removal of the carbide by hydrogenation at 150°-194° C. and reexposure of the sample of synthesis gas, a synthesis rate of 250-300 x 10^{-4} grams carbon per gram cobalt per hour was observed, an increase of 400 percent. It may be noted that the distribution of synthesis products was about the same in both cases.

In another case, a sample of cobalt-thoria-kieselguhr (100:18:100) catalyst which had been completely carbided at 210° C. was found to exhibit no appreciable activity in the synthesis under conditions where a control sample, similarly reduced initially but not carbided, gave copious yields of oil and water. Partial hydrogenation of the carbided sample at 190° C., with removal of 72 percent of the carbon which had been introduced, restored the activity to about one-half the normal value.

It was further shown that the loss of synthesis activity on carbiding can persist even when synthesis is continued for several days. Another sample of cobalt-thoria-kieselguhr catalyst was carbided for 24 hours at 200° C. after initial reduction. On exposure to flowing synthesis gas at 180° C., the carbided sample originally showed no catalytic activity, as measured by the fractional decrease in gas volume (contraction) as the gas passed through the converter. During operation for one day at 180° C., the contraction rose to 20 percent, but it did not increase further during another day of synthesis. Increasing the temperature to 190° C. increased the contraction only to 32 percent even after a period of a day. Subsequent hydrogenation of the sample at 210° C. and reexposure to synthesis gas resulted in a normal contraction of 60 percent at 180° C. Recarbiding of the

69/ Hofer, L. J. E., and Peebles, W. C., Preparation and X-ray Diffraction Studies of a New Cobalt Carbide: Jour. Am. Chem. Soc., vol. 69, 1947, pp. 893-899.

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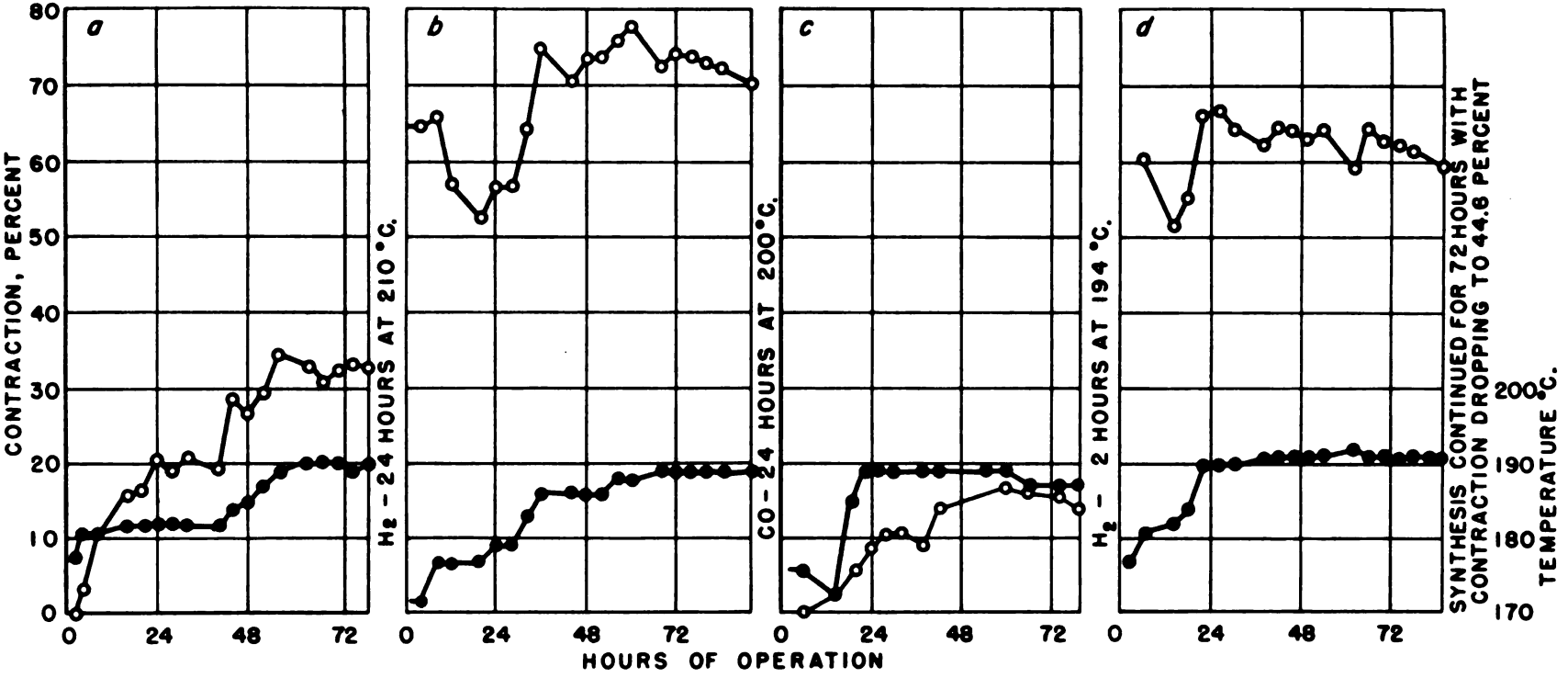

Figure 48. - Apparent contraction o and temperature of operation ● versus time for test 73 with cobalt catalyst 108B at atmospheric pressure. Catalyst was reduced in hydrogen at 360° C. and carbided with carbon monoxide at 200° C. for 24 hours prior to testing.

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active catalyst for 24 hours at 200° C. again reduced the activity to the low value observed after the first carbiding, and rehydrogenation of the carbided sample again restored the normal activity. These results are graphically presented in figure 48.

It should be noted that this inhibitory effect of precarbiding before synthesis is not explainable as a poisoning due to free carbon deposited during the carbiding, since the effect is completely reversed by hydrogenation at temperatures where free carbon is not affected. These experiments show that bulk cobalt carbide is not a catalyst for the synthesis; this conclusion, furthermore, is contrary to that which would be expected if bulk carbide were an intermediate in the synthesis.

A second line of evidence which indicates that bulk cobalt carbide is not of importance in the synthesis is furnished by X-ray analysis of catalysts which have been used in the synthesis. It has previously been shown that on low-temperature carburization with carbon monoxide, reduced cobalt catalysts form a carbide, Co_2C, which has a characteristic X-ray diffraction pattern. This fact provides a means of identifying cobalt carbide in the catalyst. It was then observed by X-ray analysis that if catalyst samples were partly carbided before synthesis and then used in synthesis, carbide was still present in the samples, even after several days of synthesis. (In pure hydrogen, the carbide would have been completely removed as methane in several hours.) On the other hand, if the samples were used in the synthesis immediately after reduction (that is, no precarbiding), no carbide was ever found at the conclusion of the synthesis. In other words, bulk carbide is apparently not built up in appreciable quantity during steady-state synthesis, while carbide already present apparently is not removed at an appreciable rate during steady-state synthesis. Although quantitative determinations of the amount of carbide present under given conditions have yet to be made, the indication is that bulk carbide is neither formed nor destroyed as part of the synthesis process.

The final evidence to be adduced in connection with the role of bulk carbide is also based on X-ray analysis. It has been shown that cobalt in catalysts reduced at 360°-400° C. is present primarily as cubic cobalt. Cubic cobalt is thermodynamically unstable with respect to hexagonal cobalt at temperatures below about 360° C. Carbiding of the reduced sample and hydrogenation of the carbide, both at 200° C., always convert the cobalt to the low-temperature stable, hexagonal form. Now all samples of cobalt catalysts (not precarbided) which have been examined after use in the synthesis have given a diffraction pattern identical with that of the freshly reduced catalyst. If any appreciable amount of the cobalt had been through the cycle of carbiding and hydrogenation at any time during the synthesis, it would have appeared as hexagonal, not cubic, cobalt in the used catalyst sample. Since this did not occur, it follows that, within the limits of the X-ray method, the cobalt was not converted to bulk carbide and subsequently hydrogenated during the synthesis; that is, bulk carbide is not intermediate in the synthesis.

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Synthesis of Liquid Fuels by Hydrogenation of Coal

Experiments in Small Autoclaves

Data obtained in the absence of high-pressure hydrogen show that solvents for bituminous coal may be grouped into three classes: The very good solvents 1, 2, 3, 4-tetrahydro-5-hydroxynaphthalene and O-cyclohexylphenol; the moderately effective solvent tetralin; and the less effective solvents dicyclohexyl, 2-methoxy-1-cyclohexylbenzene, diphenyl, naphthalene, cresol, and o-phenylphenol. This classification of solvents on the basis of their effectiveness can be correlated with the chemical structure of the solvents. The least effective group of solvents can dissolve 20 to 30 percent of the coal at 400° C. Apparently the only requirement for this action is that the solvent be a high-boiling aromatic compound or a hydroaromatic compound that is only slowly dehydrogenated under these conditions. If the solvent can function as a good hydrogen donor, its effectiveness is enhanced. Tetralin is easily dehydrogenated and accordingly gives moderately good liquefaction results. If, in addition to a hydroaromatic ring, a solvent possesses an aromatic hydroxyl group, this solvent is an extremely effective vehicle for liquefying coal. Such solvents are o-cyclohexylphenol and 1, 2, 3, 4-tetrahydro-5-hydroxynaphthalene.

In the group of less-effective solvents, the hydroaromatic compounds are generally superior to the completely aromatic compounds. The effectiveness of tetralin and the other hydroaromatic solvents is almost certainly due to their ability to act as hydrogen donors to the coal substance. In pure compound studies, it has been shown 70/ that tetralin can readily hydrogenate anthracene. It was of considerable interest in the present instance to learn the extent of the hydrogen transfer from o-cyclohexylphenol to coal. At the conclusion of one solvation experiment, the cyclohexylphenol-phenylphenol fraction was separated from the reaction mixture by distillation at atmospheric pressure and the ultraviolet absorption spectrum of this mixture determined. The spectra showed the conversion of 9 percent o-cyclohexylphenol to o-phenylphenol which corresponded to a hydrogen consumption of 1.4 percent by the dry, ash free coal. This small consumption of hydrogen was very effective in producing liquefaction of the coal.

The effectiveness of a small quantity of hydrogen available from a hydrogen donor like tetralin is illustrated by another series of solvation experiments. In this group of experiments the solvent consisted of a mixture of tetralin and cresol in varying proportions. In each experiment, 160 grams of solvent mixture was heated with 40 grams of coal for 0.5 hour at 400° C. The results are shown in figure 49. These results show that the addition of small quantities of cresol to tetralin or the addition of small quantities of tetralin to cresol results in higher liquefaction than that obtained with the pure solvent alone. The addition of a small amount of tetralin to cresol appears to have a particularly great effect. This effect can be ascribed to the influence of the small but effective quantity of hydrogen furnished by the tetralin.

70/ Orchin, M., Hydrogenation of Anthracene by Tetralin: Jour. Am. Chem. Soc., vol. 66, 1944, pp. 535-538.

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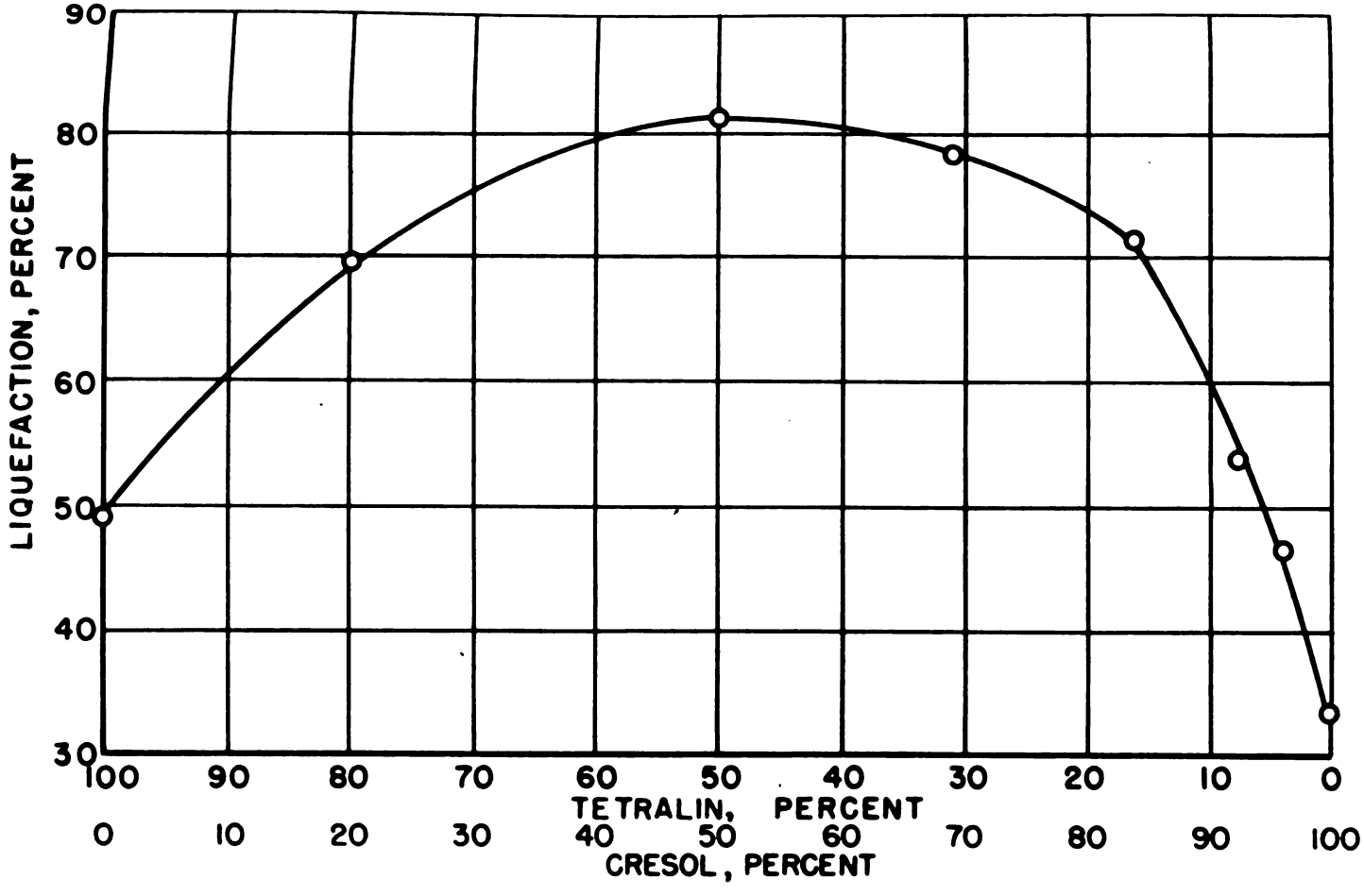

Figure 49. - Solvation experiments with various proportions of tetralin and cresol.

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The effectiveness of a small quantity of hydrogen in producing coal liquefaction is probably due to the hydrogenolysis of some peculiarly susceptible linkages in the coal structure. Thermal treatment alone does not sever these bonds to produce a liquid product; but if the coal is made to consume about 1 to 2 percent hydrogen, a completely liquid product can be obtained. The hydrogenolyzed bonds are very probably carbon-to-oxygen bonds.

The effect of an hydroxyl group in the solvent is indeed striking. 1, 2, 3, 4-tetrahydronaphthalene gives about 50 percent liquefaction; 1, 2, 3, 4-tetrahydro-5-hydroxynaphthalene gives 85 percent liquefaction. The methyl ether of cyclohexylphenol (o-methoxycyclohexylbenzene) gives 29 percent liquefaction; o-cyclohexylphenol gives 82 percent liquefaction. The effectiveness of the hydroxyl group is probably related to its hydrogen bonding properties.

The effect of concentration of the hydroxylated solvent in a mixture used for the solvation of coal is shown in table 31. In these experiments, a mixture of various concentrations of o-cyclohexylphenol in the methyl ether of o-cyclohexylphenol was used as the vehicle. The results show that a large excess of the hydroxylated solvent must be present to obtain good liquefaction of coal.

TABLE 31. - Solvation experiments at 400° C. (0.5 hour), using various concentrations of O-cyclohexylphenol (R-OH) in 2-methoxy-1-cyclohexylbenzene ($R\text{-}O\text{-}CH_3$)

Vehicle	Liquefaction, percent
100 percent R-OH	81.6
38 percent R-OH; 62 percent $R\text{-}OCH_3$	50.2
8 percent R-OH; 92 percent $R\text{-}OCH_3$	32.6
100 percent $R\text{-}OCH_3$	30.2

The solutions of coal in cyclohexylphenol are quite stable. In one experiment such a solution was kept in air at 45° for 19 days. During this time, virtually no material precipitated. The word "solution" is used in the sense that no material can be separated from the solutions by ordinary centrifugation or filtration. Microscopic examination of these solutions at 400 magnification showed them to be substantially free of solid particles. It is conceivable that the "solutions" are colloidal dispersions.71/

In the presence of high-pressure hydrogen, the degree of liquefaction obtained with hydroaromatic compounds was substantially the same as that with the corresponding aromatic compound. Under solvation conditions, however, the hydroaromatic compound in each instance gave superior results. It is apparent that, under solvation conditions, a hydrogen-donor vehicle is superior to a completely aromatic vehicle, while, under hydrogenation

71/ Kreulen, D. J. W.; Fuel: Vol. 24, 1946, p. 99. This is an article of special interest in connection with the colloidal nature of coal.

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conditions, the high-pressure hydrogen serves as the principal source of hydrogen, and the hydroaromatic feature of structure of the vehicle is no longer essential for good liquefaction results. The higher consumption of hydrogen observed with the aromatic partner of each pair was due to hydrogenation of the aromatic vehicle. That the hydroaromatic feature of structure is without significance in hydrogenation experiments is also shown by a comparison of results with cresol under solvation and hydrogenation conditions. The presence of high-pressure hydrogen increased the liquefaction from 32.1 percent to 91.7.

Although under hydrogenation conditions it does not appear to make much difference whether an aromatic, a hydroaromatic, or a naphthenic compound is used, considerable advantage is to be gained from using material as a vehicle that contains an aromatic hydroxyl group.

Detailed data have been obtained on the efficiency of zinc-antimony and tin-zinc-antimony mixtures as catalysts in the conversion of bituminous coal to heavy oil. In the presence of tin and ammonium chloride, which is the catalyst usually employed for liquefaction of bituminous coal, the production of benzene-soluble material is quite rapid, but the conversion of this material to a petroleum ether-soluble oil is a slow reaction. This is illustrated by figure 50 from which it is seen that the rate of disappearance of asphaltic material (benezene-soluble, petroleum ether-insoluble) is slow at temperatures below about 450° C. The efficiency of new catalysts in accelerating conversion of asphaltic materials to petroleum ether-soluble oil is best-studied at temperatures below 450° C. In figure 51, the efficiencies at 415° C. of 1 percent tin metal, 1 percent zinc-antimony alloy, and 0.5 percent tin metal plus 0.5 percent of zinc-antimony alloy are shown. It is apparent that tin is a good liquefaction catalyst but a relatively poor one in accelerating asphalt conversion, whereas the reverse is true for zinc-antimony alloy, which is a poor liquefaction catalyst but which greatly accelerates asphalt conversion. The ternary catalyst containing tin, and zinc-antimony alloy is very efficient for both liquefaction and asphalt conversion.

Separation and Identification of Constituents of Synthetic Liquid Fuels

The steps in the separation and identification of synthetic liquid-fuel components are: Primary distillation; separation of distillation fractions into types of compounds by solvent extraction or by adsorption on alumina or silica gel; precision distillation; analysis of lower-boiling constituents by spectrometric methods; identification of higher-boiling constituents by measurements of density; refractive index; and preparation of pure derivatives of known melting point. The laboratories in which these procedures are conducted are the precision distillation laboratory shown in figure 52, the spectrometric laboratory, figure 53, and the organic laboratory, figure 54.

The limitations of atmospheric pressure distillation were clearly recognized as a result of work on the characterization of light oil from coal hydrogenation.72/

72/ Kaplan, E. H., Storch, H. H., and Orchin, M., Hydrogenation and Liquefaction of Coal. Part V. Characterization of Light Oil: Bureau of Mines Tech. Paper 690, 1946, 18 pp.

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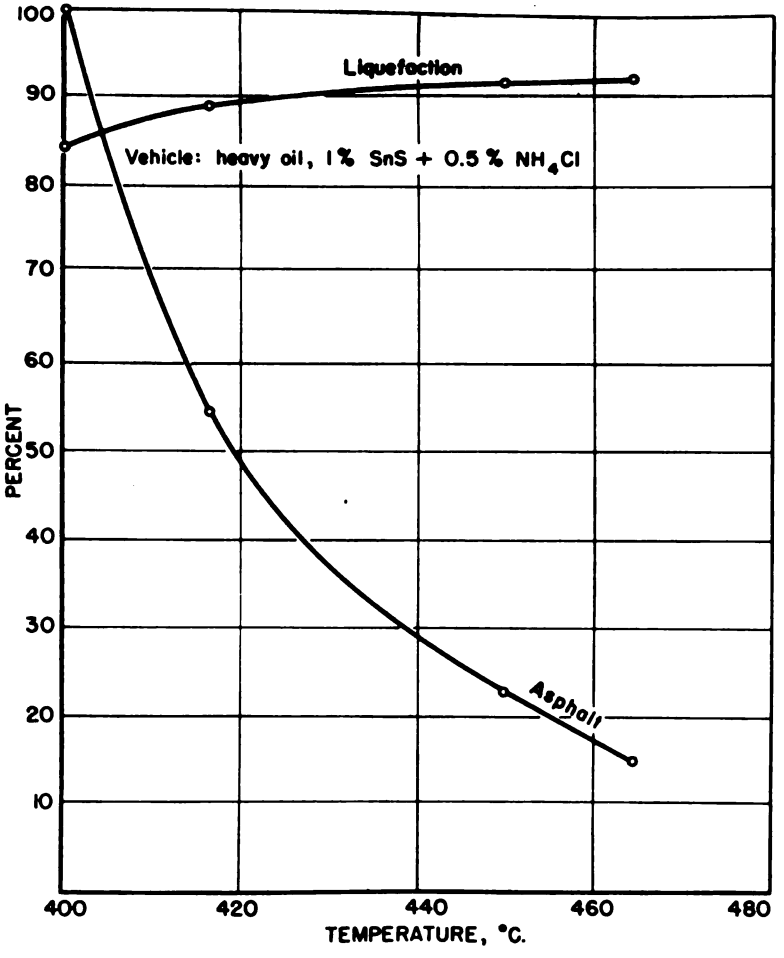

Figure 50. - Effect of temperature on liquefaction and asphalt formation.

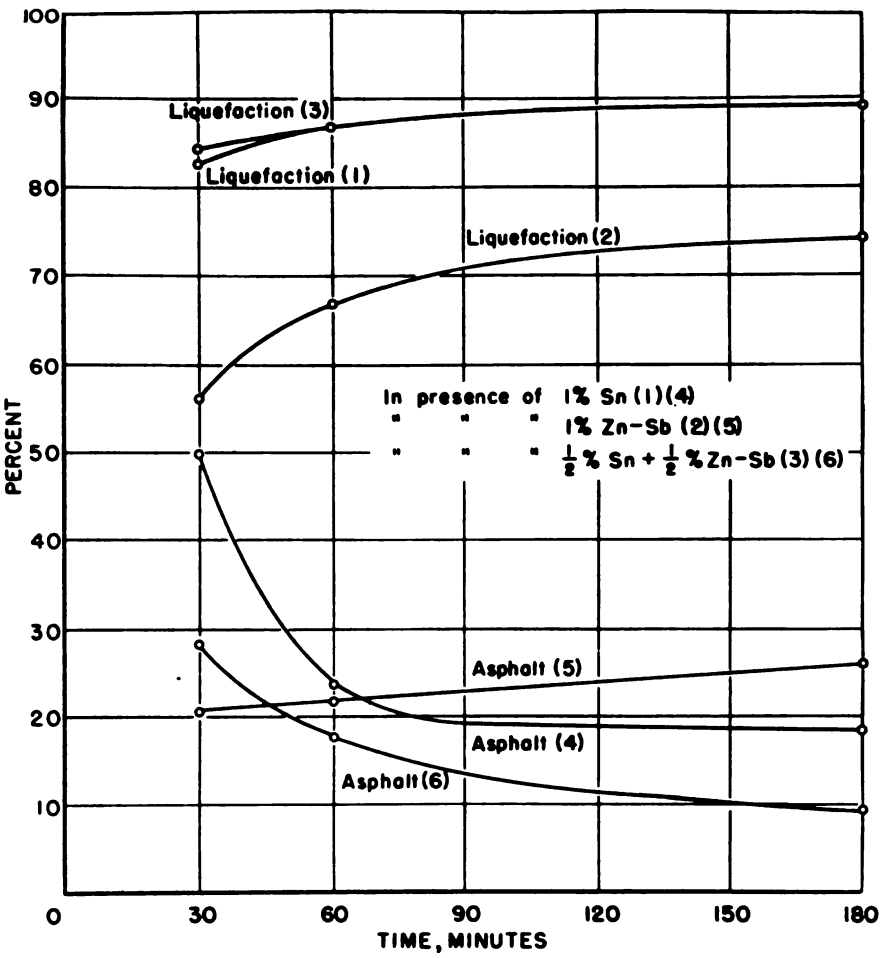

Figure 51. - Liquefaction of coal and asphalt formation at 415° C.

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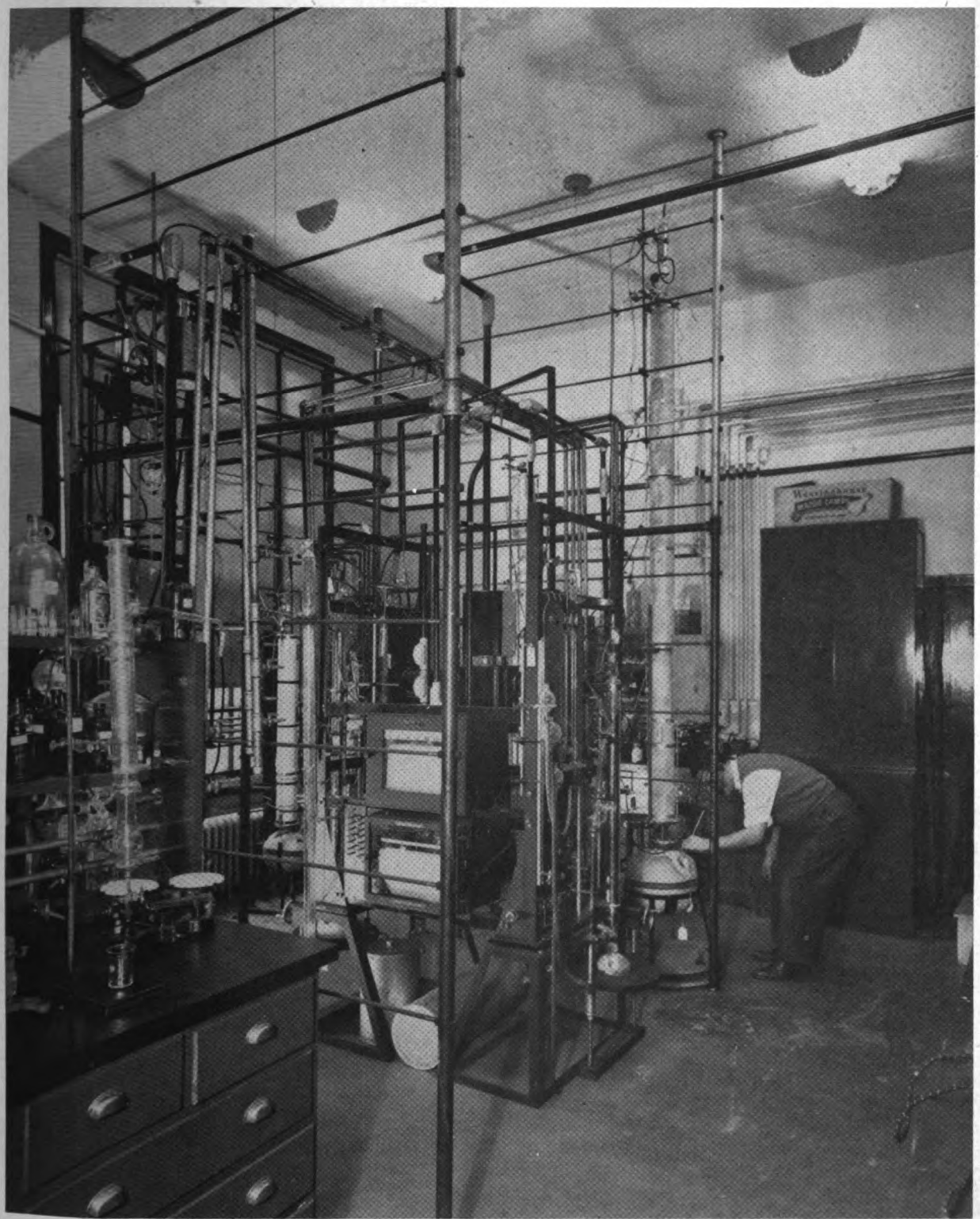

Figure 52. - Precision distillation laboratory.

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Figure 53. - Spectrometric laboratory.

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Figure 54. - Organic chemistry laboratory.

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For the characterization of high-boiling oils from both the Fischer-Tropsch and the coal-hydrogenation processes, precise vacuum distillation is essential. To establish satisfactory standards for vacuum distillation, several pairs of compounds whose boiling point and other physical properties differ only to a small degree are being investigated. Cyclohexyl-cyclopentane plus n-dodecane constituted one pair and another was tridecane plus dicyclohexyl.73/ Because a study of the vapor pressures of the pure compounds and of mixtures showed that these two pairs were not entirely satisfactory as standards for precision vacuum distillation, further work is planned on the study of pairs consisting of hydrocarbons and the corresponding deutrocarbons.

For the identification of narrow boiling fractions containing only one type of hydrocarbon, their characteristic physical constants, namely, melting points, refractive indices, infra-red74/ and ultra-violet absorption spectra, are determined. In addition, the preparation of chemical derivatives, such as the complex compounds of aromatics with 2-4-7-trinitrofluorenone,75/ is useful for identification purposes.

Identification of compounds in coal hydrogenation oils and an understanding of the probable mode of formation has been aided by a study of aromatic cyclodehydrogenation of substituted aromatic compounds.76,77/

Recent precision distillation and infra-red spectrometric work on Fischer-Tropsch products showed that all of the straight-chain paraffins up to those containing 20 carbon atoms were present in the distillable fraction (about 85 percent of the total product). About 10 percent of branched-chain paraffins, chiefly those with a methyl group in the 1 or 2 position, was present.

A new and very promising X-ray diffraction procedure for determining the extent of branching of Fischer-Tropsch products using not more than 1 cc. of sample is being developed. All normal aliphatic organic compounds, such as hydrocarbons, alcohols, and fatty acids in the liquid state, have molecular diameters (or "Bragg" spacings, as determined from the diffraction pattern) between 4.54 and 4.64 Angstrom units, with the exception of the

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73/ Orchin, M., and Feldman, J., The Isomerization of Dicyclohexyl: Jour. Am. Chem. Soc., vol. 68, December 1946, pp. 2737-2738.

74/ Friedel, R. A., McKinney, D. S., The Infra-red Absorption Spectra of Liquid and Gaseous Morpholine: Jour. Am. Chem. Soc., vol. 69, 1947, pp. 604-607.

75/ Orchin, M., and Woolfolk, E. O., Molecular Complexes with 2-4-7-trinitrofluorenone: Jour. Am. Chem. Soc., vol. 68, 1946, pp. 1727-1729, vol. 69, 1947, pp. 1225-1227.

76/ Orchin, M., Synthesis of Fluorene and Its Derivatives: U. S. Patent 2,414,118, January 1947.

77/ Orchin, M., and Reggel, L., Aromatic Cyclodehydrogenation, Part V. A Synthesis of Fluoranthene: Jour. Am. Chem. Soc., vol. 69, 1947, pp. 505-509.

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first three or four members of the series. Branched compounds, such as 2, 2, 4-trimethylpentane, have Bragg spacings of 6.00 Ångstrom units; less highly branched hydrocarbons having intermediate spacings. The Bragg spacings of straight-chain and branched chain hydrocarbons were plotted against the ratio of the numbers of carbon atoms in the branched chains to the total number of carbon atoms. The data are well-represented by a smooth curve, which will be used as a standard for determining the degree of branching of Fischer-Tropsch products which are available in only very small amounts from catalyst testing experiments.

Chemical Engineering Research, Design of Special Instruments and Equipment, and Cost Estimates on Synthetic Liquid Fuel Processes

Experiments to Provide Data for the Design of Fischer-Tropsch Pilot Plants

The data necessary for (mathematical as opposed to "intuitive") design of Fischer-Tropsch pilot plants are: A knowledge of the chemical nature of the rate-determining step in the synthesis, the temperature and pressure coefficients of the rate, complete data on the composition of the reacting system at several points along the path of the gases through the catalyst bed, the heat evolved during the synthesis, and adequate knowledge of the laws governing heat transfer in the particular process being considered. Some of these data, such as the temperature and pressure coefficients of the synthesis rate, have been obtained from recent measurements in the physical chemistry and catalyst-testing laboratories. Proof of the nature of the rate-determining step is not yet available. For the internally cooled Fischer-Tropsch converter, analysis of the composition of the reacting system at several points in the 8-foot catalyst bed is underway. Selection and improvement of the mathematical analysis procedures also are being conducted.

An analytical criterion was published[78] for the choice of individual components in systems of many constituents and relations derived[79] which form the basis for systematic calculation of the equilibrium composition of such systems as, for example, the equilibrium obtained in production of synthesis gas. These calculations are also of value in the study of the composition of the Fischer-Tropsch reacting system at intervals along the catalyst bed. Expressions were derived[80] for the temperature distribution in an adiabatic catalytic converter, as, for example, in the hot-gas recycle process.

Fuels and Lubricants Missions

Although the major portion of the work of the Fuels and Lubricants Missions to Germany was completed before the end of June 1946, five men remained

78/ Brinkley, S. R., Jr., Note on the Conditions of Equilibrium for Systems of Many Constituents: Jour. Chem. Phys., vol. 14, 1946, pp. 563-564.

79/ Brinkley, S. R., Jr., Calculation of the Equilibrium Composition of Systems of Many Constituents: Jour. Chem. Phys., vol. 15, 1947, pp.107-110.

80/ Brinkley, S. R., Jr.,Heat Transfer Between a Fluid and a Porous Solid Generating Heat: Jour. Appl. Phys., vol. 18, 1947, pp. 582-585.

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or were sent to Germany during 1947 to direct the activities of several groups of German fuels and lubricants technologists who are leading experts in their fields. These technologists have been compiling summary reports of progress in hydrogenation of coal, synthesis of carbon monoxide and hydrogen by the Fischer-Tropsch process, production of synthetic lubricants, and use of additives for improving the performance of motor fuels. Some of their reports are now being edited by the Joint Intelligence Objectives Agency. Others are now available from the Publications Board, United States Department of Commerce, and because of the broad scope of the investigations and the interest in the formation some reports have been published by the Bureau of Mines.[81/82]

Samples of catalysts, fuels, and lubricants were distributed for analysis and report by the various members of the Sample Analysis Groups working under the joint sponsorship of the American Petroleum Institute and the Bureau of Mines.

The library of Foreign Synthetic Liquid Fuels information has been moved from Washington to the Bureau of Mines at Pittsburgh, Pa. Arrangements have been continued with the Library of Congress for providing copies of microfilm reels to those who cannot conveniently make use of the library facilities. There are now 1,375 separate reports prepared by 11 different groups, all relating to some phase of synthetic liquid fuels and their production.

81/ Faragher, W. F., and Horne, W. A., Interrogation of Dr. Pier and Staff I. G. Farbenindustrie A. G., Ludwigshafen and Oppau: Bureau of Mines Inf. Circ. 7376, 1946, 27 pp.

82/ Holroyd, R., Report on Investigations by Fuels and Lubricants Teams at the I. G. Farbenindustrie, A. G., Works, Ludwigshafen and Oppau: Bureau of Mines Inf. Circ. 7375, 1946, 75 pp.

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